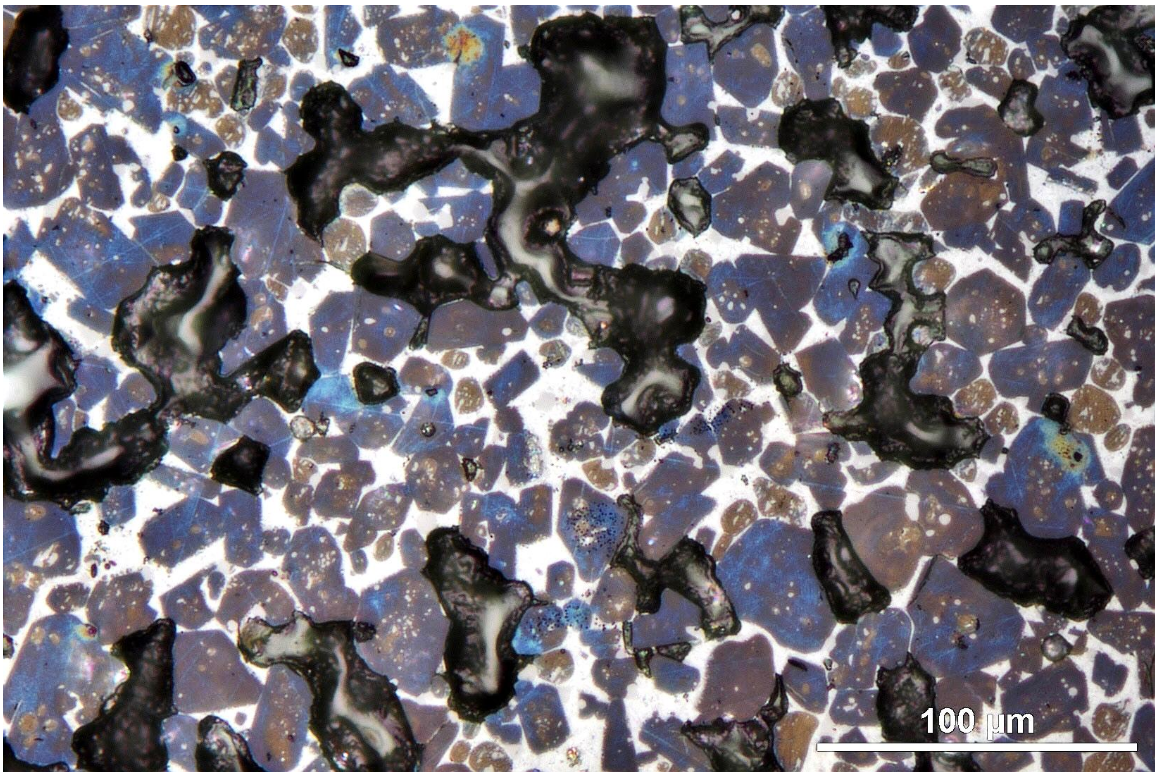
NIST Special Publication 260-204

Certification of Standard Reference Material® 2686b
Portland Cement Clinker



Paul Stutzman
Laura Mundy
Alan Heckert

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National Institute of Standards and Technology
U.S. Department of Commerce

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Paul Stutzman
Laura Mundy
Materials and Structural Systems Division
Engineering Laboratory

Alan Heckert
Statistical Engineering Division
Information Technology Laboratory

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April 2021

U.S. Department of Commerce
Gina M. Raimondo, Secretary

National Institute of Standards and Technology
James K. Olthoff, Performing the Non-Exclusive Functions and Duties of the Under Secretary of Commerce for Standards and Technology & Director, National Institute of Standards and Technology

National Institute of Standards and Technology Special Publication 260-204
Natl. Inst. Stand. Technol. Spec. Publ. 260-204, 48 pages (April 2021)
CODEN: NSPUE2

This publication is available free of charge from:
https://doi.org/10.6028/NIST.SP.260-204

Abstract

A new Standard Reference Material® (SRM) for portland cement clinker has been produced for the Office of Standard Reference Materials at the National Institute of Standards and Technology (NIST). The SRM clinkers are intended for use in developing and testing quantitative methods of phase analysis for portland cement and cement clinker. The new SRM is one of three clinkers available representing the range of grain sizes and compositions of North American clinker production.

SRM 2686b is a medium-grained, heterogeneous phase distribution clinker similar in phase composition and grain size to the original SRM 2686. Certification of phase abundance was accomplished using quantitative x-ray powder diffraction and scanning electron microscopy with image analyses. These methods provide mutually unique means to establish phase abundance, which subsequently are combined to establish certified values and uncertainties. This clinker differs from the earlier SRMs by the presence of α-C_2S. Since SEM imaging does not distinguish between the α and β polymorphs of C_2S, a certified value representing the combined mass fraction is provided and informational values are provided for the individual polymorph fractions. For alkali sulfates and free lime, phase abundance is established using a single method (XRD) so only informational values are provided.

While the XRD data are close to that of microscopy, some distinct differences are seen. The disagreements may reflect the difficulty in resolving the fine-grained aluminate and ferrite interstitial phases using the microscope and challenges in decomposing highly overlapped powder diffraction data. The XRD data do display greater precision than replicate measurements by microscopy, likely the result of the specimen homogenization resulting from grinding the clinker to a powder.

The certified reference values are consensus values, calculated by combining the results from both measurement techniques using the DerSimonian-Laird method with the standard uncertainties based upon the Horn-Horn-Duncan variance estimate. Reference values are best estimates based upon a single measurement technique. Reference values are provided for phases periclase, arcanite, aphthitalite, and lime from only the XRD data.

Key words

Cement clinker, consensus means, image analysis, microscopy, quantitative analysis, Rietveld analysis, x-ray powder diffraction.

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1. Introduction

The Standard Reference Material® (SRM) clinkers are used by industry for developing and validating methods of quantitative phase analysis. Portland cement clinker is produced by heating a mixture of limestone and shale in a cement kiln to temperatures approaching 1400 ˚C, which is subsequently ground to a fine powder with additions of gypsum and limestone to produce portland cement. Clinker is composed of a set of crystalline phases that react with water to form the hydration products that bind the aggregates in portland cement concrete. Knowledge of the types and amounts of crystalline components of clinker is critical in monitoring quality control in clinker production to produce cements conforming to specific cement Types according to ASTM-International specifications.

The certified reference values represent consensus means and uncertainties based upon two independent analytical methods, quantitative x-ray powder diffraction (XRD) and image analysis of scanning electron microscope (SEM) image sets. The three SRM clinkers exhibit a range of grain sizes from a very coarse-grained, a medium-grained, and a fine-grained clinker and range of phase compositions. Clinkers in the 2686 series are selected for their medium-grained crystal size relative to the other clinkers, a heterogeneous phase distribution with localized nests of free lime and belite. Alite occurs as subhedral to anhedral crystals approximately 30 µm in size. Belite occurs in large clusters with an approximate crystal size of 20 µm. The matrix, or interstitial phases, are comprised of fine-grained but differentiated ferrite and aluminate. Periclase crystals are disseminated throughout the microstructure ranging from a few micrometers to about 10 micrometers in size and the alkali sulfate phases are disseminated throughout the microstructure.

2. Material Selection, Sampling and Processing

2.1. Clinker Selection

Initial screening to identify if a supply was suitable involved obtaining a 1 kg grab sample of clinker from the Cemex Victorville California plant that produced the original SRM clinker, preparing epoxy-embedded polished sections for light and electron microscopy, and grinding a subsample for x-ray powder diffraction. Because product consistency is a principal objective in cement production and because the raw materials remained similar, it was anticipated that textural aspects of the clinker and phase types and amounts would remain similar to that of the original SRM material. The clinker selected for SRM 2686b contains medium-grained silicates with streaks and nests of belite along with a fine-grained interstitial matrix consisting of aluminate and ferrite. Periclase crystals as 1 µm to 10 µm equant inclusions scattered throughout the clinker and alkali sulfates occurring along grain boundaries. The fresh clinker sample grain size was consistent with previous 2686 clinker specimens, making this material suitable as a new SRM to fill the medium-grained clinker position.

One container (No. 1115) was used to assess the potential for an amorphous phase fraction. About 4 g of sample was crushed and micronized for 8 minutes and then blended with annealed and disaggregated fluorite as an internal standard. Fluorite is a suitable internal standard because its mass attenuation coefficient (≈ 91 cm^2/g) is close to that of the clinker

(≈ 101 cm^2/g) and produces relatively few, strong diffraction peaks. Analysis of three replicate scans with sample repacking indicated that the amorphous content of the clinker was negligible (0.1 % ± 0.7 %). This finding is consistent with the conventional thought of a glassy phase being unlikely in clinker because of its relatively slow cooling [1].

2.2. Clinker Sampling

400 kg of raw clinker was received from the cement plant for processing and packaging. The as-received clinker was distributed across a large tray and examined to remove any foreign materials. The entire volume of material was subsampled by sieving to retain nodules of a narrow size range between +4 mm and -15 mm [Fig. 1]. This was performed to 1) retain nodules experiencing a similar thermal history, 2) eliminate contaminants through sieving, and 3) provide an initial sample homogenization. The SRM may not be representative of the plant production but rather to a sampling of a restricted range of production to provide a consistent sample with respect to phase types and abundance.

The next step was to reduce the nodule size into fragments that would be useful for both microscopy and for XRD analysis. A jaw crusher was used to stage-crush the clinker to the desired size interval, a process where the crushed material is sieved, capturing the size fraction between 3 mm and 4 mm, while the −3 mm material is discarded, and the +4 mm over-sized materials re-crushed until no material remained on the 4 mm sieve. The process was repeated until the entire lot of clinker had been processed. The recovery rate of the desired 3 mm to 4 mm size fraction was approximately 25 %.

This material was stored in air-tight plastic bags in sealed plastic drums and transferred to the Standard Reference Materials staff for homogenization and packaging into approximately 1200 containers each containing about 50 g of clinker, is the base unit size. This represents a change in the packaging to a larger single-container unit that will be more useful for laboratories that have different sample size requirements. Packaged containers were randomly selected for analysis with 25 containers for XRD and 12 containers for SEM with image analysis (SEM/IA).

Figure 1 Crushed and sieved clinker fragments range in size between 2 mm and 5 mm.

3. Data Collection and Analysis

3.1. Light Microscopy

Microscopy using reflected white light is a routine procedure in industry to examine clinker microstructure to assess production conditions and to quantify the phase abundance by point-count analysis. Clinker is vacuum-saturated with a low viscosity resin which is subsequently cured, and the specimen is cut and polished using a series of successively finer grit diamond polishes (6 µm down to 0.25 µm) to expose a cross section of the clinker fragments. To enhance contrast of the constituents, the polished surface may be etched using a 0.1 M potassium hydroxide solution for 30 s followed by an isopropanol rinse and rapid air drying to turn the tricalcium aluminate gray followed by a 90 second Nital ((1 ml of nitric acid (HNO_3) in 99 ml of methanol to turn the silicates blue (alite) and tan (belite). Information on etching techniques for cement clinker may be found in Campbell [2]. A detailed description on sample preparation for microscopy and for XRD may be found in [3, 4].

This clinker exhibits a medium grain size characteristic with heterogeneous phase distribution [Fig. 2] similar to that of the original SRM clinker. As shown in a set of polished, etched surfaces as viewed using a reflected light microscope, this clinker is characterized by streaks and clusters of small grain size belite, occasional free lime, small grain size alite, and a fine-grained, differentiated matrix. Periclase crystals are disseminated throughout the matrix and occasional free lime and alkali sulfates are present.

3.2. X-ray Powder Diffraction

Phase composition was assessed using quantitative x-ray powder diffraction analysis following ASTM C1365, with typical powder diffraction patterns and phase identification shown in Figures 3 and 4. Since the clinker fragments are relatively large for the purpose of microscopy, they must be ground for XRD analysis. A ground specimen maximizes the number of particles in the analyzed volume, improves powder homogeneity and packing characteristics, reduces the propensity for preferred orientation, and minimizes microabsorption that may bias diffraction pattern intensities.

Each pattern was analyzed using the Rietveld method [5], a least-squares refinement to minimize the difference between a measured x-ray powder diffraction pattern and a calculated pattern based upon crystal structure, instrument and specimen effects. The addition of the refinement of structural effects now allows accommodation for the influences of chemical and structural variability on the diffraction pattern including peak shape, peak positions, and relative intensities, and the data collection error of specimen displacement, reflected as pattern shift.

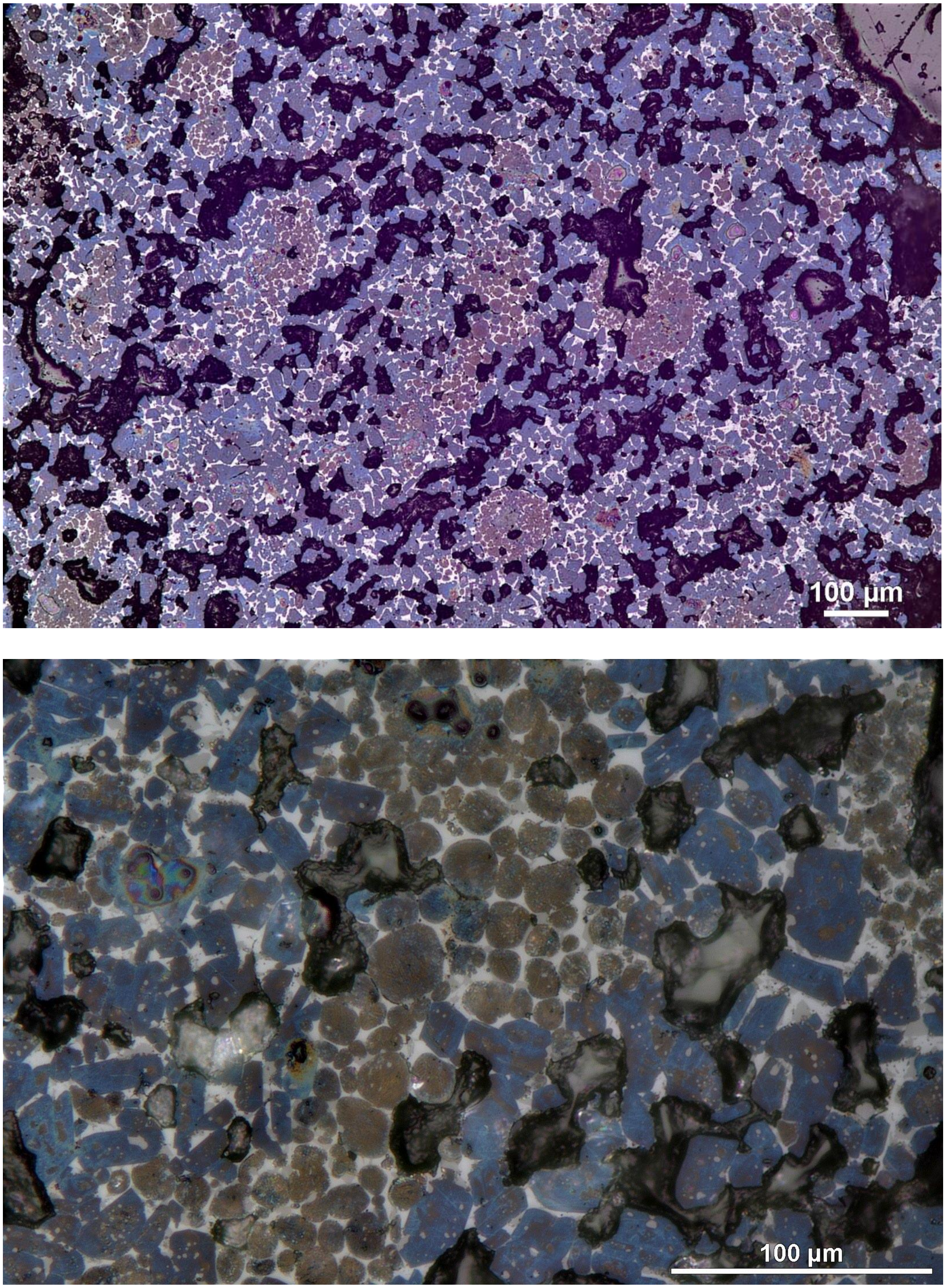


Figure 2 Reflected light microscope images of a polished, Nital-etched section of SRM 2686b shows the porous and heterogeneous distribution of the phases.

3.3. Sample Preparation for XRD

Clinker fragments are first crushed to sub-millimeter particles using a large fused alumina mortar and pestle. The crushed material is then ground in a mill to produce a median particle size around 10 µm. This orbital mill uses a set of agate cylinders as grinding elements, with 5 g of crushed clinker and 15 mL of 200-proof ethanol as a grinding lubricant. After a 6-minute grinding period, the slurry is vacuum filtered using a 50 mm #2 filter and Buchner funnel by dispensing it onto the filter followed by two rinse cycles using about 20 mL of ethanol and about 15 s of additional agitation in the mill to remove all solids. The ethanol generally dispenses clear on the second rinse indicating all the sample has been deposited on the filter. The suspension is vacuum filtered to remove all liquid and then dried in an oven at 80 ˚C. Once the sample is dry, it is disaggregated and homogenized using a small fused alumina mortar and pestle and the homogenized is powder sealed in a capped glass vial and stored in a vacuum desiccator for analysis.

3.3.1. Selective Extractions

Two selective extractions are used to concentrate different phase groups to facilitate phase identifications (Fig. 3). The potassium hydroxide – sucrose (KOHS) extraction dissolves the matrix phases tricalcium aluminate and ferrite, leaving an insoluble residue of alite, belite (β and α forms) and periclase. The KOHS extraction was performed only during material screening on one clinker container to more closely examine the silicate fraction but was not performed quantitatively. The presence of the α-form of belite was observed in this fraction by the presence of diffraction peaks at 31.8 º2θ and 33.0 º2θ.

The second selective extraction uses a salicylic acid – methanol (SAM) solution to dissolve the silicates (alite, belite) to produce a concentrated insoluble residue comprised of tricalcium aluminate, ferrite, periclase and alkali sulfates and eliminating the diffraction pattern interference from the silicates. Refinement using both cubic and orthorhombic forms of tricalcium aluminate indicates the orthorhombic form predominates, so it will be used for subsequent analyses. The SAM extraction is performed quantitatively for each clinker sample (Appendix A) so a second set of composition data are provided to estimate the concentrations of the interstitial phases on a whole-clinker basis. More details on selective extractions may be found in [3, 4] and an example on how the quantitative data from the SAM residue and bulk clinker are combined into a single test result is provided in Table 1.

3.4. Scanning Electron Microscopy

Specimen preparation involved crushing clinker fragments into particles of about 250 µm in diameter in an attempt to provide a more homogeneous specimen for imaging. About 2 g of clinker was gently crushed using a mortar and pestle and sieved to capture the fines. Any remaining coarse material was placed back in the mortar and crushed and sieved, repeating the process until no coarse fragments remained. The crushed powder was blended with epoxy to make a thick paste, which was pressed into a cylinder mold and allowed to cure at room temperature overnight followed to a final cure step at 60 °C for about 2h to complete the epoxy polymerization [Fig. 4].

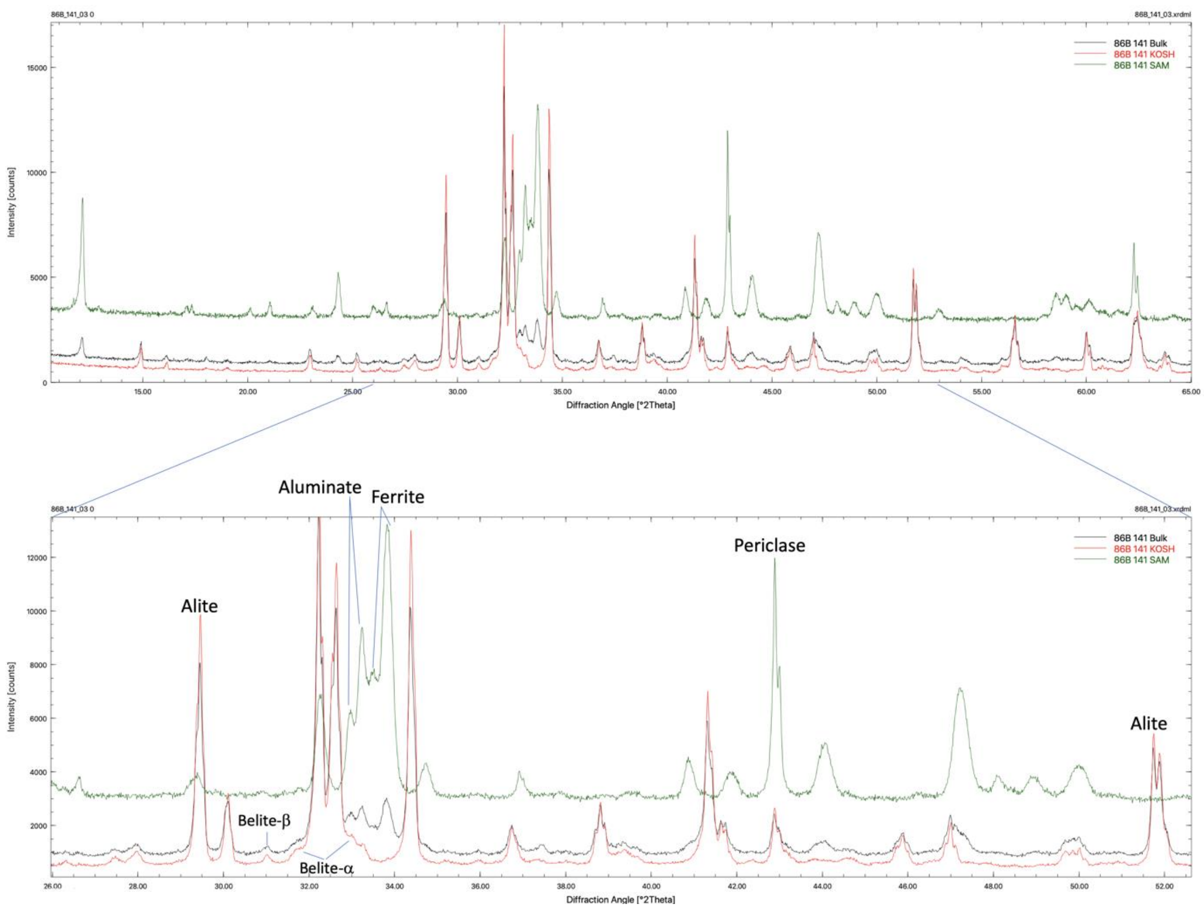


Figure 3 X-ray powder diffraction patterns of the selective extraction residues with an expanded view (lower) showing how selective dissolution enhances the detection limits for qualitative identifications.

Table 1 Combining bulk clinker (B) and recalculated SAM IR data (Sr) for a more comprehensive test result. Lime is dissolved in the SAM extraction (S) and arcanite was below detection limits (nd) in the bulk clinker samples.

		Whole Clinker			Recalculated SAM IR			SAM IR (19.08 %)		
#8	Mean	B1	B2	B3	Sr1	Sr2	Sr3	S1	S2	S3
alite	63.67	62.73	64.16	64.12						
belite-β	14.64	14.90	14.90	14.41						
belite-α	2.12	2.18	2.18	2.14						
aluminate (orthorhombic)	4.03	4.43	4.43	3.99	3.86	3.96	4.01	20.25	20.75	21.01
ferrite	11.36	11.20	11.52	11.14	11.52	11.45	11.37	60.40	60.00	59.58
periclase	3.53	3.69	3.69	3.57	3.52	3.50	3.51	18.40	18.37	18.40
arcanite	0.18	nd	nd	nd	0.18	0.17	0.20	0.92	0.89	1.04
lime	0.55	0.88	0.88	0.43	nd	nd	nd	nd	nd	nd

The cured block was cut to expose the clinker grain cross sections using 120 grit silicon carbide abrasive paper, smoothed using successively finer grades of silicon carbide paper down to 1200 grit. Polishing using an automated polishing instrument used diamond slurries and low-nap polishing plates with grits of 6 µm, 3 µm, 1 µm, and 0.25 µm to produce a smooth, grinding defect-free surface for imaging.

A set of registered (aligned) backscattered electron and x-ray images were collected for 10 arbitrarily selected fields for each sample as a test result. Microscope operating settings were 12 kV and 2.5 nA with a frame scan time of about three minutes and total collection time of about 40 minutes. While crushing the grains was intended to provide a more homogeneous sampling for microscopy, the clinker crystals remained relatively large and the field to field variation was greater than the XRD replicates as will be shown subsequently. Image size of 1024 x 968 pixels in 16-bit TIF files that preserve the gross counts at each pixel (Fig. 5). The image magnification covered an 800 µm field width for a lateral resolution slightly below 1 µm which is roughly the spatial resolution of the x-ray images.

Image processing and analysis following procedures documented in [3,4] uses this set of SEM backscattered electron and x-ray images. The registered image set forms a virtual image stack that include the backscattered electron, aluminum, magnesium, potassium and sulfur, the minimum necessary to uniquely identify phases and process the image set (Fig. 6). The operator designates regions typical of each phase on a stack of images and the classification algorithm seeks to group every pixel into the class that it most likely belongs [6]. To be consistent with the XRD mass fraction determinations, the area fractions must be recalculated using phase density, as described in [3].

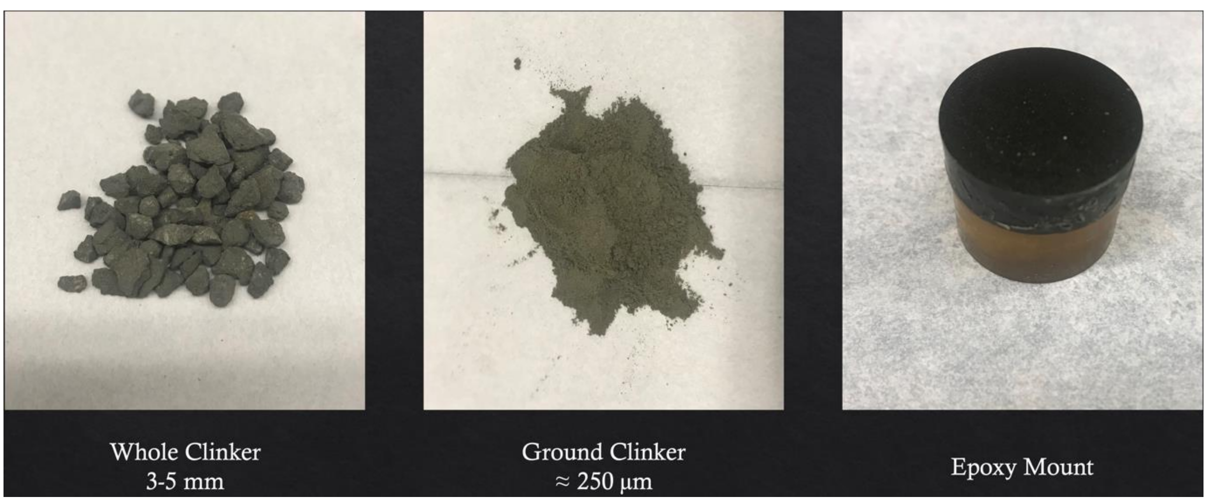


Figure 4 Transforming the clinker fragments into a crushed powder and an epoxy-embedded polished cross section provides a specimen for scanning electron microscopy and image analysis.

3.5. Image Processing and Analysis

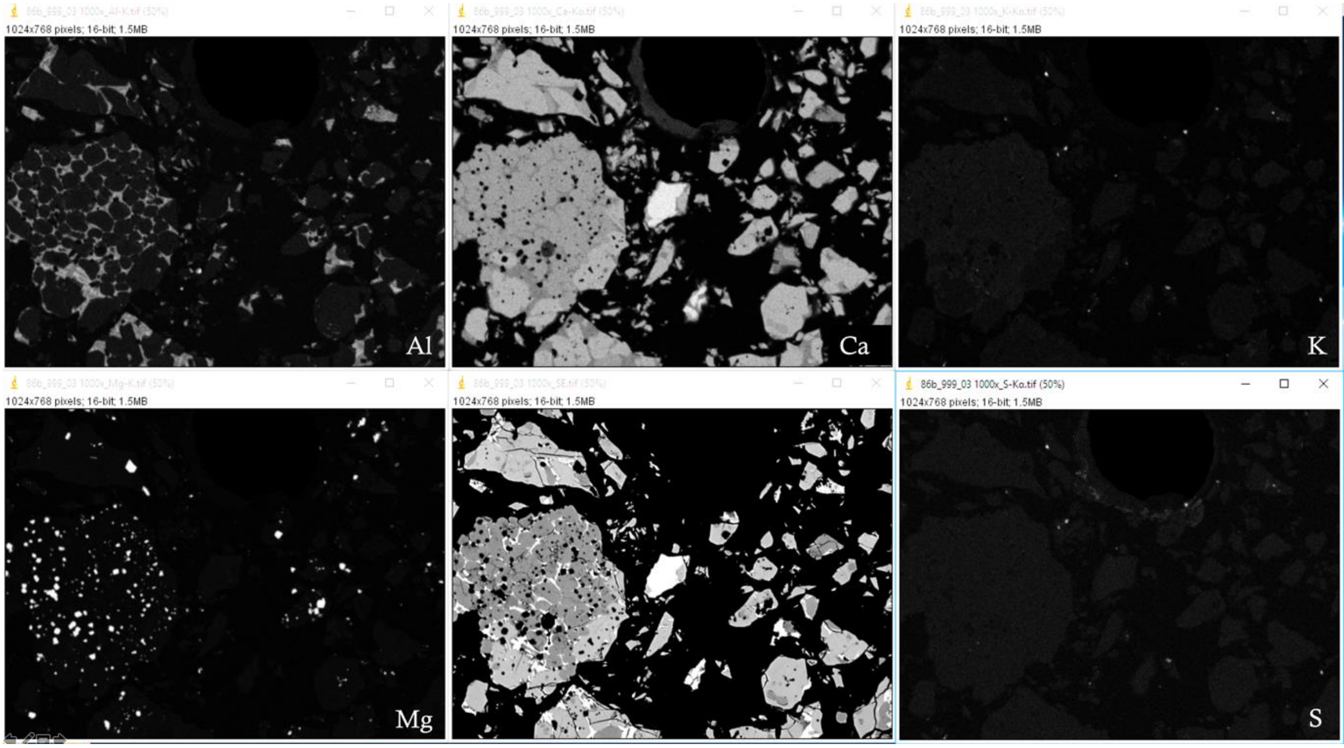


Figure 5 SEM Image set with an approximate field width of 800 um showing (top, left to right) aluminum, calcium, and potassium distribution and (bottom) magnesium, backscattered electron image and sulfur.

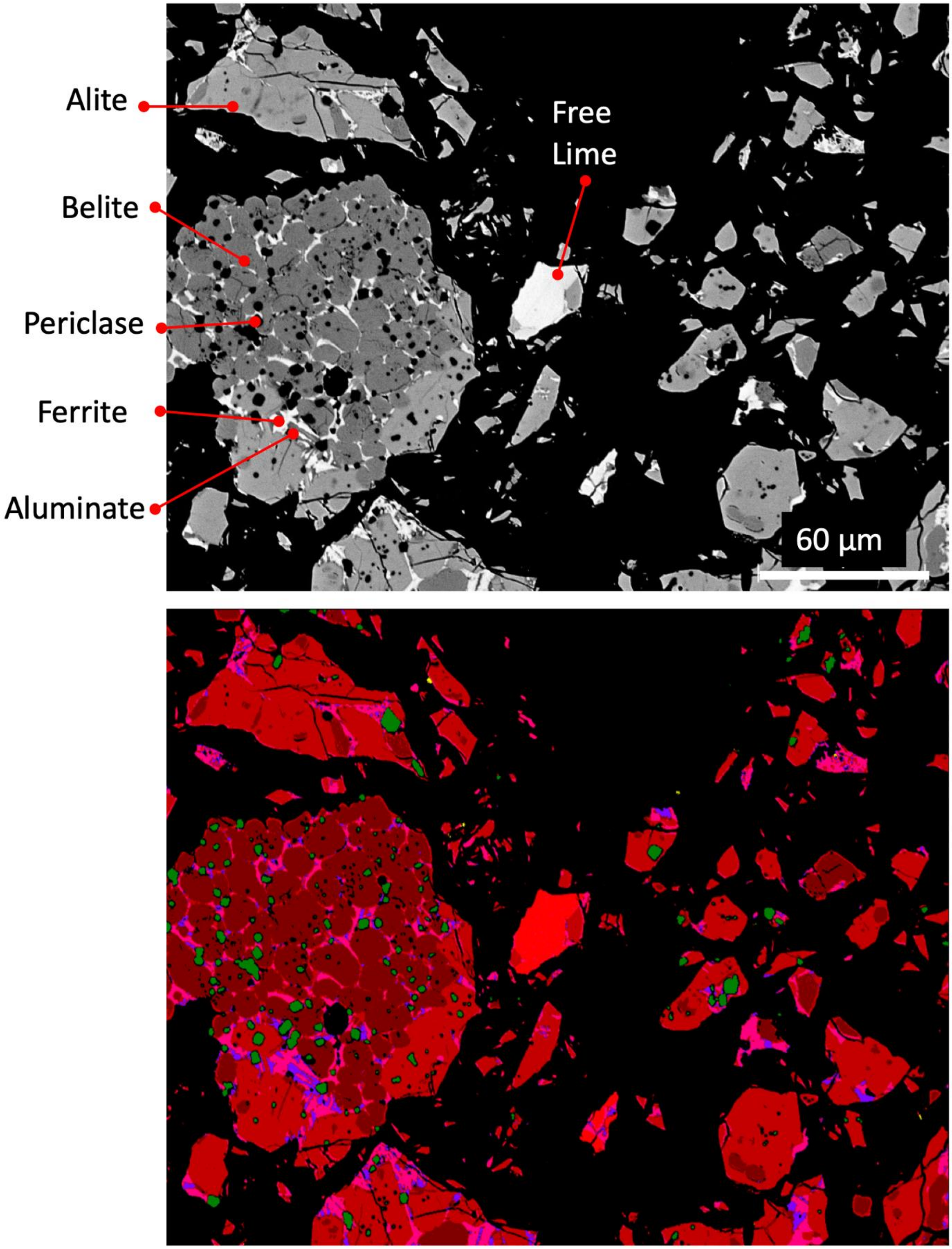


Figure 6 SEM backscattered electron (upper) and combined BE, Al, and Mg image (lower) after processing and ready for multispectral classification showing the individual phases more clearly based upon the x-ray element distribution and color.

4. Consensus Means and Uncertainties

A variety of methods are available for estimating consensus means and the associated uncertainties [7]. The grand mean is simply the average of all the data from all methods. This does not take into account either within method variability or between method variability. The mean of means is an early consensus method as an unweighted mean of method means. While this approach takes between method variability into account, it does not include within method variability. The Graybill-Deal method is a weighted mean where the weights are determined by the within method variability. However, this method does not take between method variability into account.

4.1. Certified Values

The DerSimonian-Laird (DSL) [8] and the Vangel-Rukhin (VR) [9] approaches take both the within method variance and the between method variance into account. The Vangel-Rukhin method is essentially the maximum likelihood (ML) approach. Although ML approach has excellent statistical properties, these properties are asymptotic and we have relatively few methods. The DSL approach starts with the Graybill-Deal estimate, but then adds a correction to account for the between method variability. The DSL approach can be used for either a small number of methods or a large number of methods. Our primary reason for choosing DSL over Vangel-Rukhin is that we have a small number of methods.

DSL uncertainties are computed in the following three ways: 1) as given in the original DerSimomnian-Laird paper; 2) utilizing a bootstrap method; and 3) using the Horn-Horn-Duncan (HHD) method (10) as described in Rukhin (11). The HHD uncertainties are slightly more conservative for these data than the other two methods, so we chose to use the HHD uncertainties.

Plots for each phase are presented in Figures 6 through 12, with code 1 and 2 being the two different XRD processing codes. The consensus means and associated uncertainties for the various consensus methods described in [7] are plotted in the left-hand portion while the data for XRD method 1 (Code 1) and XRD method 2 (Code 2) and SEM analyses are shown in the right-hand portion of each plot. The HHD variances were slightly more conservative than the bootstrap variances and are the recommended values. The DSL estimates were also consistent with the Vangel-Rukhin maximum likelihood estimates. Certified values for alite, belite, aluminate, and ferrite are based upon the two test methods while reference values for periclase, arcanite, and free lime are provided based upon the x-ray powder diffraction results.

Table 2 provides the results for the multiple method data obtained using the DerSimonian-Laird estimate for the consensus mean. The standard uncertainties are based on the Horn-Horn-Duncan variance estimate.

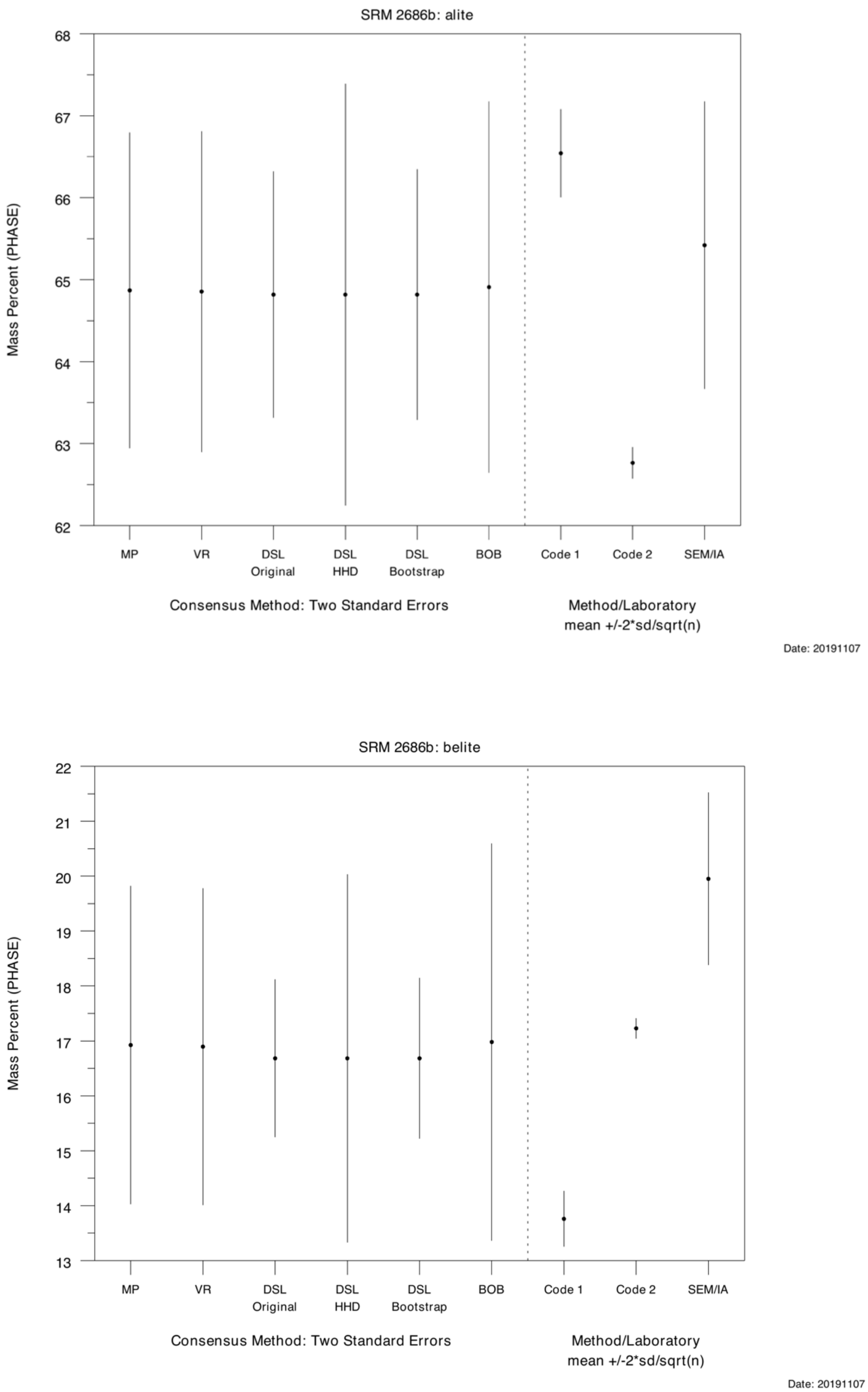
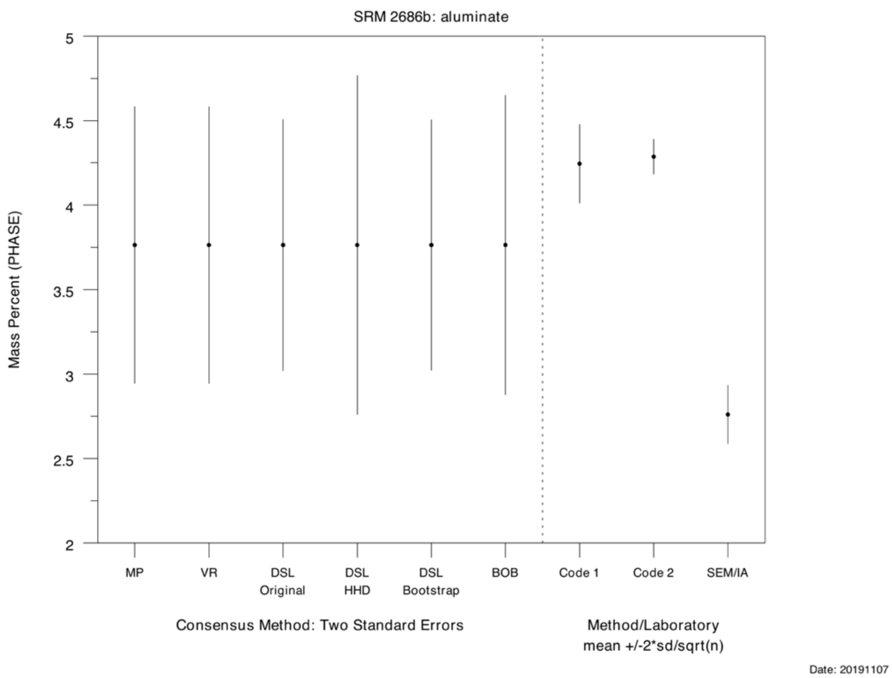


Figure 7 Consensus means expressed as mass percent by method for alite (upper) and belite (lower).



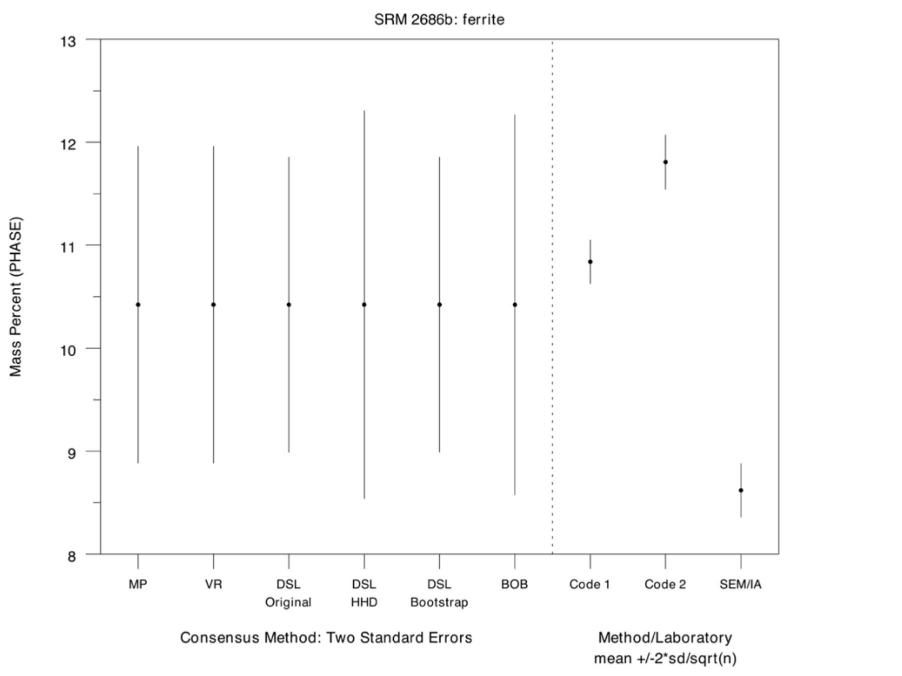
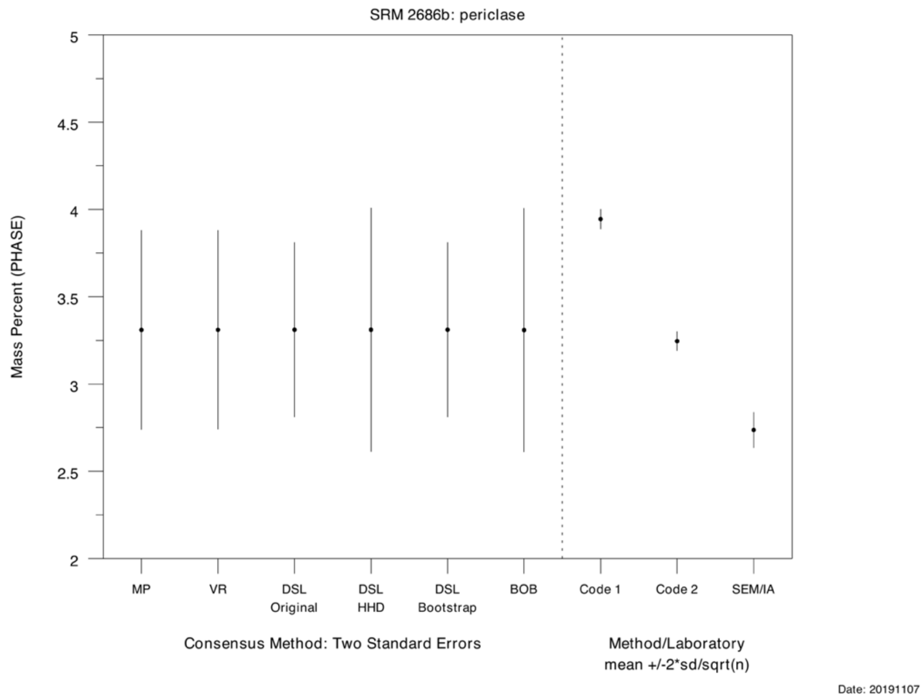


Figure 8 Consensus means expressed as mass percent by method for aluminate (upper) and ferrite (lower).



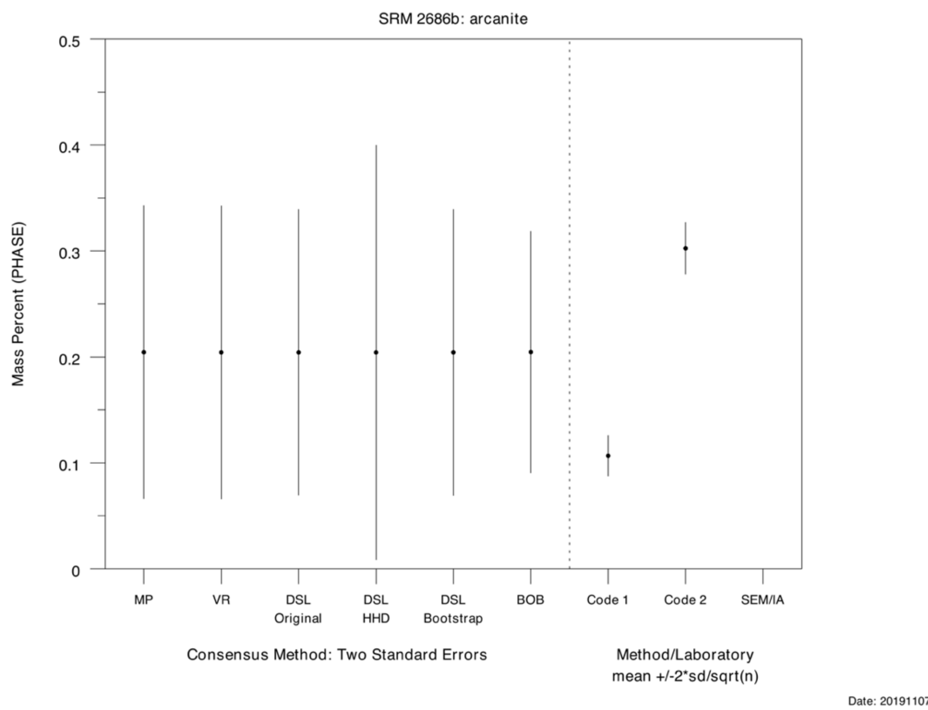


Figure 9 Consensus means expressed as mass percent by method for periclase (upper) and arcanite (lower).

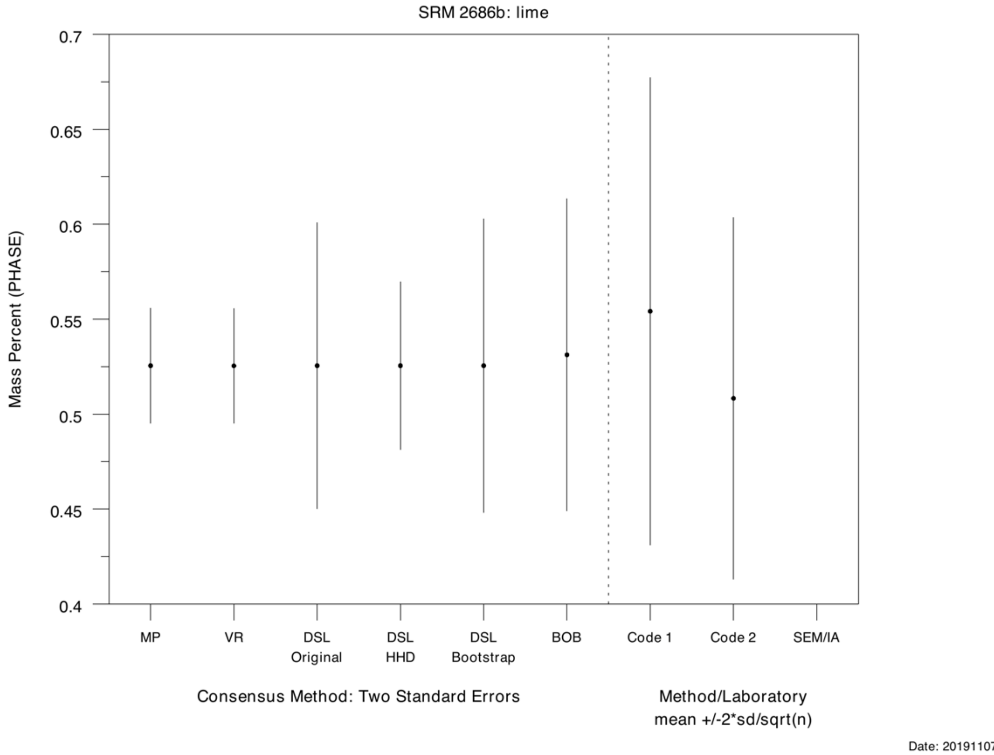


Figure 10 Consensus means expressed as mass percent by method for free lime.

Table 2 Consensus means, uncertainty and 95 % confidence limits for clinker phases.

CONSTITUENT	CONSENSUS MEAN	STANDARD UNCERTAINTY	K=2 EXPANDED UNCERTAINTY	95 % LOWER CONFIDENCE LIMIT	95 % UPPER CONFIDENCE LIMIT
ALITE	64.82	1.29	2.57	59.28	70.35
BELITE	16.68	1.68	3.35	9.47	23.89
ALUMINATE	3.76	0.50	1.00	1.60	5.92
FERRITE	10.42	0.94	1.88	6.36	14.47
PERICLASE	3.31	0.35	0.70	1.81	4.82
ARCANITE	0.20	0.10	0.20	-1.01	1.45
FREE LIME	0.53	0.02	0.08	0.24	0.81

4.2. Reference Values

Reference values represent best estimates of the true value where all known or suspected sources of bias have not been fully investigated [12] but are of interest to the user. For this cement clinker the values for arcanite and free lime as well as the bulk chemical values are based upon single methods. Free lime may hydrate upon prolonged exposure to ambient conditions but may be recovered by heat-treating the clinker specimen at a temperature of 550 °C for a period of at least one hour.

4.2.1. Bulk Chemistry

The bulk chemical composition for this clinker was measured by a single method, x-ray fluorescence analysis by an outside collaborating laboratory on four arbitrarily selected specimens. The bulk chemical data are expressed as oxides as is the convention in the cement industry, and these specimens would be useful for purposes of expanding calibration curves for specific analytes. The test results (Table 3) represent the overall mean of the four individual specimens. These data are based upon cement calibration curves using pressed pellets using NIST 1880 series SRMs and some low SO_3 standards.

Table 3 Mean values and individual replicate bulk oxide data for four clinker samples.

Vial	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**SO_3**	**Na_2O**	**K_2O**	**TiO_2**	**Cl**
73	21.17	4.16	3.67	63.64	4.35	0.02	0.25	0.45	0.23	0.0012
365	21.35	4.19	3.75	64.13	4.40	0.02	0.23	0.44	0.23	0.0012
999	21.50	4.26	3.74	63.90	4.39	0.04	0.25	0.46	0.23	0.0022
60	21.29	4.09	3.72	64.21	4.33	0.08	0.24	0.44	0.23	0.0031
Mean	**21.33**	**4.18**	**3.72**	**63.97**	**4.37**	**0.04**	**0.24**	**0.45**	**0.23**	**0.0019**

5. References

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6. Appendix A. Salicylic Acid/Methanol Selective Extraction Residue by Sample.

Clinker Sample	Residue (g)	Ground Clinker (g)	Insoluble Residue (%)
8	1.580	8.283	19.08
12	1.562	8.010	19.50
60	1.947	10.061	19.35
73	2.040	10.278	19.85
76	2.031	10.197	19.92
141	1.969	10.030	19.63
142	1.935	10.028	19.30
161	1.989	10.189	19.52
164	1.930	10.053	19.20
169	2.206	10.131	21.77
190	1.802	9.163	19.67
216	2.013	10.230	19.68
241	1.989	10.255	19.40
249	1.949	10.095	19.31
329	1.950	10.013	19.47
365	1.946	10.035	19.39
373	1.956	10.139	19.29
389	2.377	10.059	23.63
419	1.886	10.008	18.84
454	1.906	10.026	19.01
472	1.914	10.070	19.01
480	1.891	10.006	18.90
999	1.531	8.362	18.31
1039	1.953	10.008	19.51
1115	1.768	9.321	18.97
Mean, 1s			19.58, 1.04

7. Supplemental Materials

7.1. XRD Data for analysis codes 1 and 2 and SEM/IA data.

Method	Sample	Alite	Belite	Aluminate	Ferrite	Periclase	Arcanite	Lime
1	8	64.71	15.95	3.82	11.22	3.84	0.07	0.51
1	12	66.85	13.27	4.17	11.05	3.82	0.15	0.51
1	60	65.87	14.20	4.10	11.21	3.81	0.10	0.73
1	73	66.52	13.72	4.01	11.37	3.78	0.10	0.18
1	76	65.18	14.43	5.08	10.12	3.84	0.09	0.91
1	141	65.79	14.32	4.07	10.99	3.84	0.11	0.83
1	142	66.72	13.83	5.09	10.12	3.96	0.06	0.14
1	161	66.30	14.33	4.05	10.86	3.88	0.12	0.76
1	164	66.79	13.68	4.15	10.87	3.94	0.11	0.59
1	169	66.21	13.80	3.83	10.81	4.49	0.19	0.63
1	190	68.28	12.23	4.10	10.82	3.97	0.15	0.57
1	216	66.50	13.90	4.09	11.00	4.02	0.08	0.38
1	241	67.22	13.26	4.24	10.87	3.97	0.06	0.38
1	249	66.30	13.71	4.18	10.93	4.01	0.11	0.72
1	329	66.92	13.54	4.05	10.97	3.97	0.13	0.51
1	365	66.64	13.59	4.08	11.04	4.04	0.11	0.43
1	373	66.47	13.93	4.07	10.89	4.02	0.06	0.65
1	389	66.28	13.99	4.05	11.19	4.01	0.11	0.20
1	419	66.34	13.88	4.11	10.95	3.96	0.07	0.77
1	454	66.29	14.08	4.02	10.96	4.05	0.08	0.65
1	472	67.27	13.30	4.01	10.98	4.04	0.12	0.35
1	480	66.80	13.37	3.97	11.07	4.12	0.10	0.48
1	999	66.54	13.77	4.09	10.71	4.06	0.15	0.89
1	1039	66.94	13.63	4.25	10.66	3.99	0.11	0.60
1	1115	65.91	14.98	3.94	10.85	3.92	0.05	0.65
2	8	62.63	17.55	4.23	11.51	3.22	0.30	0.60
2	12	62.96	16.87	4.27	11.88	3.20	0.36	0.47
2	60	62.00	17.83	4.26	11.73	3.25	0.27	0.63
2	73	62.82	17.05	4.18	12.22	3.27	0.30	0.22
2	76	62.50	17.62	4.27	11.61	3.25	0.36	0.68
2	141	62.50	17.62	4.27	11.61	3.25	0.36	0.68
2	142	62.64	17.42	4.12	11.99	3.21	0.28	0.14
2	161	62.69	17.24	4.43	11.62	3.20	0.29	0.65
2	164	62.80	17.05	4.36	11.64	3.19	0.27	0.58
2	169	62.73	16.77	4.35	12.33	3.79	0.59	0.55
2	190	63.72	16.37	4.17	11.81	3.20	0.45	0.54
2	216	62.63	16.96	4.39	12.00	3.28	0.26	0.35

2	241	62.67	17.48	4.39	11.69	3.18	0.25	0.42
2	249	62.20	17.61	4.40	11.62	3.20	0.30	0.61
2	329	62.87	17.50	4.26	11.68	3.19	0.31	0.47
2	365	61.98	18.08	4.22	11.80	3.22	0.34	0.41
2	373	62.85	16.92	4.43	11.61	3.23	0.28	0.55
2	389	62.96	16.91	4.61	13.09	3.52	0.41	0.24
2	419	62.67	17.28	4.24	11.51	3.15	0.29	0.65
2	454	62.68	17.61	4.21	11.42	3.19	0.31	0.56
2	472	63.59	16.78	4.04	11.66	3.19	0.29	0.38
2	480	63.58	16.56	4.24	11.54	3.25	0.26	0.43
2	999	63.13	16.97	4.08	11.18	3.14	0.31	0.80
2	1039	62.99	17.09	4.29	11.71	3.19	0.27	0.62
2	1115	62.60	17.67	3.95	11.61	3.28	0.27	0.59
3	60	69.33	15.89	2.96	8.64	2.85		
3	73	63.45	21.97	2.45	9.40	2.70		
3	76	69.46	16.19	2.54	8.96	2.82		
3	141	66.50	18.86	2.65	9.13	2.82		
3	329	64.25	21.32	2.68	8.61	2.69		
3	365	64.65	20.53	3.37	8.40	2.68		
3	373	67.50	18.88	2.71	8.26	2.66		
3	419	61.19	22.77	3.00	8.21	2.80		
3	454	65.36	20.27	2.69	8.34	2.98		
3	480	62.52	22.83	2.56	8.25	2.37		

7.2. SEM/Image Analysis Summary.

SEM/IA results expressed as mass fractions for n=10 field of view per sample

Sample	Alite	Belite	Aluminate	Ferrite	Periclase
60	69.33	15.89	2.96	8.64	2.85
73	63.45	21.97	2.45	9.40	2.70
76	69.46	16.19	2.54	8.96	2.82
141	66.50	18.86	2.65	9.13	2.82
329	64.25	21.32	2.68	8.61	2.69
365	64.65	20.53	3.37	8.40	2.68
373	67.50	18.88	2.71	8.26	2.66
419	61.19	22.77	3.00	8.21	2.80
454	65.36	20.27	2.69	8.34	2.98
480	62.52	22.83	2.56	8.25	2.37

7.3. Consensus Means Summary by Phase
7.3.1. SRM 2686b Compound: Alite


```
            Consensus Means Analysis
               (Full Sample Case)

Data Summary:
Response Variable: Y1
Lab-ID Variable: METHOD
Number of Observations:                                  34
Grand Mean:                                   0.6487971E+02
Grand Standard Deviation:                     0.2268653E+01
Total Number of Labs:                                     3
Minimum Lab Mean:                             0.6276500E+02
Maximum Lab Mean:                             0.6654333E+02
Minimum Lab SD:                               0.3352475E+00
Maximum Lab SD:                               0.2773920E+01
Mean of Lab Means:                            0.6490978E+02
SD of Lab Means:                              0.1940351E+01
SD of Lab Means (wrt to grand mean):          0.1940700E+01
Within Lab (pooled) SD:                       0.1606823E+01
Within Lab (pooled) Variance:                 0.2581879E+01

            Table 1: Summary Statistics by Lab
```

Lab ID	n(i)	Mean	Variance	Standard Deviation	Standard Deviation of the Mean
1	12	0.6654333E+02	0.8682061E+00	0.9317758E+00	0.2689805E+00
2	12	0.6276500E+02	0.1123909E+00	0.3352475E+00	0.9677763E-01
3	10	0.6542100E+02	0.7694632E+01	0.2773920E+01	0.8771905E+00

```
 1. Method: Mandel-Paule
    Estimate of (unscaled) Consensus Mean:  0.6486927E+02
    Estimate of (scaled) Consensus Mean:    0.5569302E+00
    Between Lab Variance (unscaled):        0.3708883E+01
    Between Lab SD (unscaled):              0.1925846E+01
    Between Lab Variance (scaled):          0.2598021E+00
    Standard Deviation of Consensus Mean:   0.9634094E+00
    Standard Uncertainty (k = 1):           0.9634094E+00
    Expanded Uncertainty (k = 2):           0.1926819E+01
    Expanded Uncertainty (k =  1.9599640):  0.1888248E+01
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.6298102E+02
    Upper 95% (normal) Confidence Limit:    0.6675752E+02
    Note: Mandel-Paule Best Usage:
          6 or More Labs:


 3. Method: Vangel-Rukhin Maximum Likelihood
    Estimate of (unscaled) Consensus Mean:  0.6485526E+02
```

```
    Estimate of (scaled) Consensus Mean:    0.5532225E+00
    Between Lab Variance (unscaled):        0.2631987E+01
    Between Lab SD (unscaled):              0.1622340E+01
    Between Lab Variance (scaled):          0.1843670E+00
    Standard Deviation of Consensus Mean:   0.9787831E+00
    Standard Uncertainty (k = 1):           0.9787831E+00
    Expanded Uncertainty (k = 2):           0.1957566E+01
    Expanded Uncertainty (k =  1.9599640):  0.1918380E+01
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.6293688E+02
    Upper 95% (normal) Confidence Limit:    0.6677364E+02
    Note: Vangel-Rukhin Maximum Likelihood
          Best Usage: 6 or More Labs


4a. Method: DerSimonian Laird (original variance)
    Estimate of Consensus Mean:               0.6481803E+02
    Estimate of Variance of Consensus Mean:  0.5643579E+00
    Estimate of Between Lab Variance:         0.1469013E+01
    Standard Uncertainty (k = 1):             0.7512376E+00
    Expanded Uncertainty (k = 2):             0.1502475E+01
    Degrees of Freedom:                                   2
    t Percent Point Value:                    0.4302653E+01
    Lower 95% (t-value) Confidence Limit:     0.6158571E+02
    Upper 95% (t-value) Confidence Limit:     0.6805034E+02
    Note: DerSimonian-Laird Best Usage:
          Any Number of Labs:


4b. Method: DerSimonian Laird - Horn-Horn-Duncan Variance
     Estimate of Consensus Mean:               0.6481803E+02
     Estimate of Variance of Consensus Mean:  0.1653938E+01
     Estimate of Between Lab Variance:         0.1469013E+01
     Standard Uncertainty (k = 1):             0.1286055E+01
     Expanded Uncertainty (k = 2):             0.2572111E+01
     Degrees of Freedom:                                   2
     t Percent Point Value:                    0.4302653E+01
     Lower 95% (t-value) Confidence Limit:     0.5928458E+02
     Upper 95% (t-value) Confidence Limit:     0.7035148E+02
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


4d. Method: DerSimonian Laird - Bootstrap Variance
     Number of Bootstrap Samples                                   100000
     Estimate of Consensus Mean:                              0.6481803E+02
     Estimate of Variance of Consensus Mean:                  0.5845488E+00
     Standard Uncertainty (k = 1):                            0.7645579E+00
     Expanded Uncertainty (k = 2):                            0.1529116E+01
     Lower 95% (percentile bootstrap) Confidence Limit:  0.6332277E+02
     Upper 95% (percentile bootstrap) Confidence Limit:  0.6631470E+02
     Lower 95% (symmetric bootstrap) Confidence Limit:   0.6332136E+02
     Upper 95% (symmetric bootstrap) Confidence Limit:   0.6631470E+02
     K (symmetric bootstrap) Coverage Factor:            0.1957566E+01
     Lower 95% (kernel bootstrap) Confidence Limit:      0.6331342E+02
     Upper 95% (kernel bootstrap) Confidence Limit:      0.6632062E+02
     K (kernel bootstrap) Coverage Factor:               0.1965304E+01
```

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

11. Method: BOB (Bound on Bias)

Estimate of Consensus Mean:	0.6490978E+02
Within Lab Uncertainty:	0.3075313E+00
Between Lab Uncertainty:	0.1090711E+01
Standard Uncertainty (k = 1):	0.1133237E+01
Expanded Uncertainty (k = 2):	0.2266474E+01
Lower 95% (k = 2) Confidence Limit:	0.6264330E+02
Upper 95% (k = 2) Confidence Limit:	0.6717625E+02

Note: BOB Best Usage:
5 or Fewer Labs:

Table 2: 95% Confidence Limits

Method	Consensus Mean	Lower Limit	Upper Limit	Uncertainty (k*SE)
1. Mandel-Paule	0.6486927E+02	0.6298102E+02	0.6675752E+02	0.1888248E+01
3a. Vangel-Rukhin ML	0.6485526E+02	0.6293688E+02	0.6677364E+02	0.1918380E+01
4a. DerSimonian-Laird (original)	0.6481803E+02	0.6158571E+02	0.6805034E+02	0.3232314E+01
4b. DerSimonian-Laird (H-H-D)	0.6481803E+02	0.5928458E+02	0.7035148E+02	0.5533450E+01
4d. DerSimonian-Laird (perc. bootstrap)	0.6481803E+02	0.6332277E+02	0.6631470E+02	0.1496673E+01
4d. DerSimonian-Laird (symm. bootstrap)	0.6481803E+02	0.6332136E+02	0.6631470E+02	0.1496673E+01
4d. DerSimonian-Laird (kern bootstrap)	0.6481803E+02	0.6331342E+02	0.6632062E+02	0.1504611E+01
11. BOB	0.6490978E+02	0.6264330E+02	0.6717625E+02	0.2266474E+01

Table 3: Standard Uncertainties (k = 1)

Method	Consensus Mean	Standard Uncertainty (k = 1)	Relative Standard Uncertainty (%)
1. Mandel-Paule	0.6486927E+02	0.9634094E+00	0.1485155E+01
3a. Vangel-Rukhin ML	0.6485526E+02	0.9787831E+00	0.1509181E+01
4a. DerSimonian-Laird (original)	0.6481803E+02	0.7512376E+00	0.1158995E+01
4b. DerSimonian-Laird (H-H-D)	0.6481803E+02	0.1286055E+01	0.1984101E+01
4d. DerSimonian-Laird (bootstrap)	0.6481803E+02	0.7645579E+00	0.1179545E+01
11. BOB	0.6490978E+02	0.1133237E+01	0.1745865E+01

Table 4: Expanded Uncertainties (k = 2)

Method	Consensus Mean	Expanded Uncertainty (k = 2)	Relative Expanded Uncertainty (%)
1. Mandel-Paule	0.6486927E+02	0.1926819E+01	0.2970311E+01
3a. Vangel-Rukhin ML	0.6485526E+02	0.1957566E+01	0.3018362E+01
4a. DerSimonian-Laird (original)	0.6481803E+02	0.1502475E+01	0.2317989E+01
4b. DerSimonian-Laird (H-H-D)	0.6481803E+02	0.2572111E+01	0.3968203E+01
4d. DerSimonian-Laird (bootstrap)	0.6481803E+02	0.1529116E+01	0.2359090E+01
11. BOB	0.6490978E+02	0.2266474E+01	0.3491729E+01

7.3.2. SRM 2686b Compound: Belite

```
            Consensus Means Analysis
               (Full Sample Case)

Data Summary:
Response Variable: Y2
Lab-ID Variable: METHOD
Number of Observations:                              34
Grand Mean:                                0.1680500E+02
Grand Standard Deviation:                  0.2900671E+01
Total Number of Labs:                                 3
Minimum Lab Mean:                          0.1376000E+02
Maximum Lab Mean:                          0.1995100E+02
Minimum Lab SD:                            0.3251526E+00
Maximum Lab SD:                            0.2485449E+01
Mean of Lab Means:                         0.1697978E+02
SD of Lab Means:                           0.3102975E+01
SD of Lab Means (wrt to grand mean):       0.3110350E+01
Within Lab (pooled) SD:                    0.1451034E+01
Within Lab (pooled) Variance:              0.2105499E+01
```

Table 1: Summary Statistics by Lab

Lab ID	n(i)	Mean	Variance	Standard Deviation	Standard Deviation of the Mean
1	12	0.1376000E+02	0.7736727E+00	0.8795867E+00	0.2539148E+00
2	12	0.1722833E+02	0.1057242E+00	0.3251526E+00	0.9386348E-01
3	10	0.1995100E+02	0.6177454E+01	0.2485449E+01	0.7859678E+00

```
 1. Method: Mandel-Paule
    Estimate of (unscaled) Consensus Mean:  0.1692418E+02
    Estimate of (scaled) Consensus Mean:    0.5110929E+00
    Between Lab Variance (unscaled):        0.9313702E+01
    Between Lab SD (unscaled):              0.3051836E+01
    Between Lab Variance (scaled):          0.2429969E+00
    Standard Deviation of Consensus Mean:   0.1448997E+01
    Standard Uncertainty (k = 1):           0.1448997E+01
    Expanded Uncertainty (k = 2):           0.2897995E+01
    Expanded Uncertainty (k =  1.9599640):  0.2839982E+01
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.1408419E+02
    Upper 95% (normal) Confidence Limit:    0.1976416E+02
    Note: Mandel-Paule Best Usage:
          6 or More Labs:


 3. Method: Vangel-Rukhin Maximum Likelihood
    Estimate of (unscaled) Consensus Mean:  0.1689546E+02
    Estimate of (scaled) Consensus Mean:    0.5064550E+00
    Between Lab Variance (unscaled):        0.6013600E+01
    Between Lab SD (unscaled):              0.2452264E+01
    Between Lab Variance (scaled):          0.1568964E+00
    Standard Deviation of Consensus Mean:   0.1441409E+01
    Standard Uncertainty (k = 1):           0.1441409E+01
    Expanded Uncertainty (k = 2):           0.2882819E+01
    Expanded Uncertainty (k =  1.9599640):  0.2825110E+01
```

```
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.1407035E+02
    Upper 95% (normal) Confidence Limit:    0.1972057E+02
    Note: Vangel-Rukhin Maximum Likelihood
          Best Usage: 6 or More Labs


4a. Method: DerSimonian Laird (original variance)
    Estimate of Consensus Mean:              0.1668269E+02
    Estimate of Variance of Consensus Mean:  0.5166020E+00
    Estimate of Between Lab Variance:        0.1361933E+01
    Standard Uncertainty (k = 1):            0.7187503E+00
    Expanded Uncertainty (k = 2):            0.1437501E+01
    Degrees of Freedom:                                  2
    t Percent Point Value:                   0.4302653E+01
    Lower 95% (t-value) Confidence Limit:    0.1359016E+02
    Upper 95% (t-value) Confidence Limit:    0.1977523E+02
    Note: DerSimonian-Laird Best Usage:
          Any Number of Labs:


4b. Method: DerSimonian Laird - Horn-Horn-Duncan Variance
     Estimate of Consensus Mean:              0.1668269E+02
     Estimate of Variance of Consensus Mean:  0.2808752E+01
     Estimate of Between Lab Variance:        0.1361933E+01
     Standard Uncertainty (k = 1):            0.1675933E+01
     Expanded Uncertainty (k = 2):            0.3351866E+01
     Degrees of Freedom:                                  2
     t Percent Point Value:                   0.4302653E+01
     Lower 95% (t-value) Confidence Limit:    0.9471734E+01
     Upper 95% (t-value) Confidence Limit:    0.2389365E+02
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


4d. Method: DerSimonian Laird - Bootstrap Variance
     Number of Bootstrap Samples                                 100000
     Estimate of Consensus Mean:                          0.1668269E+02
     Estimate of Variance of Consensus Mean:              0.5360760E+00
     Standard Uncertainty (k = 1):                        0.7321721E+00
     Expanded Uncertainty (k = 2):                        0.1464344E+01
     Lower 95% (percentile bootstrap) Confidence Limit:  0.1524249E+02
     Upper 95% (percentile bootstrap) Confidence Limit:  0.1811332E+02
     Lower 95% (symmetric bootstrap) Confidence Limit:   0.1524249E+02
     Upper 95% (symmetric bootstrap) Confidence Limit:   0.1812290E+02
     K (symmetric bootstrap) Coverage Factor:            0.1967030E+01
     Lower 95% (kernel bootstrap) Confidence Limit:      0.1524126E+02
     Upper 95% (kernel bootstrap) Confidence Limit:      0.1812600E+02
     K (kernel bootstrap) Coverage Factor:               0.1971264E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


11. Method: BOB (Bound on Bias)
    Estimate of Consensus Mean:           0.1697978E+02
    Within Lab Uncertainty:               0.2770938E+00
    Between Lab Uncertainty:              0.1787188E+01
    Standard Uncertainty (k = 1):         0.1808541E+01
    Expanded Uncertainty (k = 2):         0.3617082E+01
    Lower 95% (k = 2) Confidence Limit:   0.1336270E+02
    Upper 95% (k = 2) Confidence Limit:   0.2059686E+02
    Note: BOB Best Usage:
          5 or Fewer Labs:
```

Table 2: 95% Confidence Limits

Method	Consensus Mean	Lower Limit	Upper Limit	Uncertainty (k*SE)
1. Mandel-Paule	0.1692418E+02	0.1408419E+02	0.1976416E+02	0.2839982E+01
3a. Vangel-Rukhin ML	0.1689546E+02	0.1407035E+02	0.1972057E+02	0.2825110E+01
4a. DerSimonian-Laird (original)	0.1668269E+02	0.1359016E+02	0.1977523E+02	0.3092533E+01
4b. DerSimonian-Laird (H-H-D)	0.1668269E+02	0.9471734E+01	0.2389365E+02	0.7210958E+01
4d. DerSimonian-Laird (perc. bootstrap)	0.1668269E+02	0.1524249E+02	0.1811332E+02	0.1440204E+01
4d. DerSimonian-Laird (symm. bootstrap)	0.1668269E+02	0.1524249E+02	0.1812290E+02	0.1440204E+01
4d. DerSimonian-Laird (kern bootstrap)	0.1668269E+02	0.1524126E+02	0.1812600E+02	0.1443305E+01
11. BOB	0.1697978E+02	0.1336270E+02	0.2059686E+02	0.3617082E+01

Table 3: Standard Uncertainties (k = 1)

Method	Consensus Mean	Standard Uncertainty (k = 1)	Relative Standard Uncertainty (%)
1. Mandel-Paule	0.1692418E+02	0.1448997E+01	0.8561701E+01
3a. Vangel-Rukhin ML	0.1689546E+02	0.1441409E+01	0.8531339E+01
4a. DerSimonian-Laird (original)	0.1668269E+02	0.7187503E+00	0.4308359E+01
4b. DerSimonian-Laird (H-H-D)	0.1668269E+02	0.1675933E+01	0.1004594E+02
4d. DerSimonian-Laird (bootstrap)	0.1668269E+02	0.7321721E+00	0.4388813E+01
11. BOB	0.1697978E+02	0.1808541E+01	0.1065115E+02

Table 4: Expanded Uncertainties (k = 2)

Method	Consensus Mean	Expanded Uncertainty (k = 2)	Relative Expanded Uncertainty (%)
1. Mandel-Paule	0.1692418E+02	0.2897995E+01	0.1712340E+02
3a. Vangel-Rukhin ML	0.1689546E+02	0.2882819E+01	0.1706268E+02
4a. DerSimonian-Laird (original)	0.1668269E+02	0.1437501E+01	0.8616719E+01
4b. DerSimonian-Laird (H-H-D)	0.1668269E+02	0.3351866E+01	0.2009188E+02
4d. DerSimonian-Laird (bootstrap)	0.1668269E+02	0.1464344E+01	0.8777625E+01
11. BOB	0.1697978E+02	0.3617082E+01	0.2130229E+02

7.3.3. SRM 2686b Compound: Aluminate

```
            Consensus Means Analysis
               (Full Sample Case)

 Data Summary:
 Response Variable: Y3
 Lab-ID Variable: METHOD
 Number of Observations:                              34
 Grand Mean:                               0.3823235E+01
 Grand Standard Deviation:                 0.7558247E+00
 Total Number of Labs:                                 3
 Minimum Lab Mean:                         0.2761000E+01
 Maximum Lab Mean:                         0.4286667E+01
 Minimum Lab SD:                           0.1816757E+00
 Maximum Lab SD:                           0.4060340E+00
 Mean of Lab Means:                        0.3764222E+01
 SD of Lab Means:                          0.8690657E+00
 SD of Lab Means (wrt to grand mean):      0.8720659E+00
 Within Lab (pooled) SD:                   0.3035625E+00
 Within Lab (pooled) Variance:             0.9215022E-01

            Table 1: Summary Statistics by Lab
```

Lab ID	n(i)	Mean	Variance	Standard Deviation	Standard Deviation of the Mean
1	12	0.4245000E+01	0.1648636E+00	0.4060340E+00	0.1172119E+00
2	12	0.4286667E+01	0.3300606E-01	0.1816757E+00	0.5244526E-01
3	10	0.2761000E+01	0.7556556E-01	0.2748919E+00	0.8692845E-01

```
  1. Method: Mandel-Paule
     Estimate of (unscaled) Consensus Mean:  0.3764036E+01
     Estimate of (scaled) Consensus Mean:    0.6574413E+00
     Between Lab Variance (unscaled):        0.7476542E+00
     Between Lab SD (unscaled):              0.8646700E+00
     Between Lab Variance (scaled):          0.3212044E+00
     Standard Deviation of Consensus Mean:   0.4098844E+00
     Standard Uncertainty (k = 1):           0.4098844E+00
     Expanded Uncertainty (k = 2):           0.8197689E+00
     Expanded Uncertainty (k =  1.9599640):  0.8033587E+00
     Normal PPF of 0.975:                    0.1959964E+01
     Lower 95% (normal) Confidence Limit:    0.2960678E+01
     Upper 95% (normal) Confidence Limit:    0.4567395E+01
     Note: Mandel-Paule Best Usage:
           6 or More Labs:


  3. Method: Vangel-Rukhin Maximum Likelihood
     Estimate of (unscaled) Consensus Mean:  0.3763971E+01
     Estimate of (scaled) Consensus Mean:    0.6573985E+00
     Between Lab Variance (unscaled):        0.4962696E+00
     Between Lab SD (unscaled):              0.7044640E+00
     Between Lab Variance (scaled):          0.2132055E+00
     Standard Deviation of Consensus Mean:   0.4099767E+00
     Standard Uncertainty (k = 1):           0.4099767E+00
     Expanded Uncertainty (k = 2):           0.8199535E+00
     Expanded Uncertainty (k =  1.9599640):  0.8035396E+00
```

```
    Normal PPF of 0.975:                       0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.2960431E+01
    Upper 95% (normal) Confidence Limit:    0.4567511E+01
    Note: Vangel-Rukhin Maximum Likelihood
          Best Usage: 6 or More Labs


4a. Method: DerSimonian Laird (original variance)
    Estimate of Consensus Mean:              0.3763911E+01
    Estimate of Variance of Consensus Mean:  0.1388212E+00
    Estimate of Between Lab Variance:        0.4084969E+00
    Standard Uncertainty (k = 1):            0.3725872E+00
    Expanded Uncertainty (k = 2):            0.7451744E+00
    Degrees of Freedom:                                  2
    t Percent Point Value:                   0.4302653E+01
    Lower 95% (t-value) Confidence Limit:    0.2160797E+01
    Upper 95% (t-value) Confidence Limit:    0.5367024E+01
    Note: DerSimonian-Laird Best Usage:
          Any Number of Labs:


4b. Method: DerSimonian Laird - Horn-Horn-Duncan Variance
     Estimate of Consensus Mean:              0.3763911E+01
     Estimate of Variance of Consensus Mean:  0.2523307E+00
     Estimate of Between Lab Variance:        0.4084969E+00
     Standard Uncertainty (k = 1):            0.5023253E+00
     Expanded Uncertainty (k = 2):            0.1004651E+01
     Degrees of Freedom:                                  2
     t Percent Point Value:                   0.4302653E+01
     Lower 95% (t-value) Confidence Limit:    0.1602580E+01
     Upper 95% (t-value) Confidence Limit:    0.5925242E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


4d. Method: DerSimonian Laird - Bootstrap Variance
     Number of Bootstrap Samples                                 100000
     Estimate of Consensus Mean:                          0.3763911E+01
     Estimate of Variance of Consensus Mean:              0.1380501E+00
     Standard Uncertainty (k = 1):                        0.3715509E+00
     Expanded Uncertainty (k = 2):                        0.7431019E+00
     Lower 95% (percentile bootstrap) Confidence Limit:  0.3037768E+01
     Upper 95% (percentile bootstrap) Confidence Limit:  0.4494614E+01
     Lower 95% (symmetric bootstrap) Confidence Limit:   0.3033208E+01
     Upper 95% (symmetric bootstrap) Confidence Limit:   0.4494614E+01
     K (symmetric bootstrap) Coverage Factor:            0.1966629E+01
     Lower 95% (kernel bootstrap) Confidence Limit:      0.3032735E+01
     Upper 95% (kernel bootstrap) Confidence Limit:      0.4496009E+01
     K (kernel bootstrap) Coverage Factor:               0.1970386E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


11. Method: BOB (Bound on Bias)
    Estimate of Consensus Mean:           0.3764222E+01
    Within Lab Uncertainty:               0.5168892E-01
    Between Lab Uncertainty:              0.4404220E+00
    Standard Uncertainty (k = 1):         0.4434448E+00
    Expanded Uncertainty (k = 2):         0.8868896E+00
    Lower 95% (k = 2) Confidence Limit:   0.2877333E+01
    Upper 95% (k = 2) Confidence Limit:   0.4651112E+01
    Note: BOB Best Usage:
          5 or Fewer Labs:
```

Table 2: 95% Confidence Limits

Method	Consensus Mean	Lower Limit	Upper Limit	Uncertainty (k*SE)
1. Mandel-Paule	0.3764036E+01	0.2960678E+01	0.4567395E+01	0.8033587E+00
3a. Vangel-Rukhin ML	0.3763971E+01	0.2960431E+01	0.4567511E+01	0.8035396E+00
4a. DerSimonian-Laird (original)	0.3763911E+01	0.2160797E+01	0.5367024E+01	0.1603113E+01
4b. DerSimonian-Laird (H-H-D)	0.3763911E+01	0.1602580E+01	0.5925242E+01	0.2161331E+01
4d. DerSimonian-Laird (perc. bootstrap)	0.3763911E+01	0.3037768E+01	0.4494614E+01	0.7307030E+00
4d. DerSimonian-Laird (symm. bootstrap)	0.3763911E+01	0.3033208E+01	0.4494614E+01	0.7307030E+00
4d. DerSimonian-Laird (kern bootstrap)	0.3763911E+01	0.3032735E+01	0.4496009E+01	0.7320986E+00
11. BOB	0.3764222E+01	0.2877333E+01	0.4651112E+01	0.8868896E+00

Table 3: Standard Uncertainties (k = 1)

Method	Consensus Mean	Standard Uncertainty (k = 1)	Relative Standard Uncertainty (%)
1. Mandel-Paule	0.3764036E+01	0.4098844E+00	0.1088949E+02
3a. Vangel-Rukhin ML	0.3763971E+01	0.4099767E+00	0.1089213E+02
4a. DerSimonian-Laird (original)	0.3763911E+01	0.3725872E+00	0.9898938E+01
4b. DerSimonian-Laird (H-H-D)	0.3763911E+01	0.5023253E+00	0.1334583E+02
4d. DerSimonian-Laird (bootstrap)	0.3763911E+01	0.3715509E+00	0.9871407E+01
11. BOB	0.3764222E+01	0.4434448E+00	0.1178052E+02

Table 4: Expanded Uncertainties (k = 2)

Method	Consensus Mean	Expanded Uncertainty (k = 2)	Relative Expanded Uncertainty (%)
1. Mandel-Paule	0.3764036E+01	0.8197689E+00	0.2177898E+02
3a. Vangel-Rukhin ML	0.3763971E+01	0.8199535E+00	0.2178427E+02
4a. DerSimonian-Laird (original)	0.3763911E+01	0.7451744E+00	0.1979788E+02
4b. DerSimonian-Laird (H-H-D)	0.3763911E+01	0.1004651E+01	0.2669167E+02
4d. DerSimonian-Laird (bootstrap)	0.3763911E+01	0.7431019E+00	0.1974281E+02
11. BOB	0.3764222E+01	0.8868896E+00	0.2356103E+02

7.3.4. SRM 2686b Compound: Ferrite

```
            Consensus Means Analysis
               (Full Sample Case)

Data Summary:
Response Variable: Y4
Lab-ID Variable: METHOD
Number of Observations:                             34
Grand Mean:                             0.1052794E+02
Grand Standard Deviation:               0.1376565E+01
Total Number of Labs:                                3
Minimum Lab Mean:                       0.8620000E+01
Maximum Lab Mean:                       0.1180667E+02
Minimum Lab SD:                         0.3681763E+00
Maximum Lab SD:                         0.4581848E+00
Mean of Lab Means:                      0.1042194E+02
SD of Lab Means:                        0.1633789E+01
SD of Lab Means (wrt to grand mean):    0.1638939E+01
Within Lab (pooled) SD:                 0.4152869E+00
Within Lab (pooled) Variance:           0.1724632E+00

            Table 1: Summary Statistics by Lab
```

Lab ID	n(i)	Mean	Variance	Standard Deviation	Standard Deviation of the Mean
1	12	0.1083917E+02	0.1355538E+00	0.3681763E+00	0.1062833E+00
2	12	0.1180667E+02	0.2099333E+00	0.4581848E+00	0.1322666E+00
3	10	0.8620000E+01	0.1717778E+00	0.4144608E+00	0.1310640E+00

```
 1. Method: Mandel-Paule
    Estimate of (unscaled) Consensus Mean:  0.1042220E+02
    Estimate of (scaled) Consensus Mean:    0.5655429E+00
    Between Lab Variance (unscaled):        0.2652168E+01
    Between Lab SD (unscaled):              0.1628548E+01
    Between Lab Variance (scaled):          0.2611726E+00
    Standard Deviation of Consensus Mean:   0.7696621E+00
    Standard Uncertainty (k = 1):           0.7696621E+00
    Expanded Uncertainty (k = 2):           0.1539324E+01
    Expanded Uncertainty (k =  1.9599640):  0.1508510E+01
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.8913687E+01
    Upper 95% (normal) Confidence Limit:    0.1193071E+02
    Note: Mandel-Paule Best Usage:
          6 or More Labs:


 3. Method: Vangel-Rukhin Maximum Likelihood
    Estimate of (unscaled) Consensus Mean:  0.1042233E+02
    Estimate of (scaled) Consensus Mean:    0.5655843E+00
    Between Lab Variance (unscaled):        0.1760614E+01
    Between Lab SD (unscaled):              0.1326881E+01
    Between Lab Variance (scaled):          0.1733768E+00
    Standard Deviation of Consensus Mean:   0.7694015E+00
    Standard Uncertainty (k = 1):           0.7694015E+00
    Expanded Uncertainty (k = 2):           0.1538803E+01
    Expanded Uncertainty (k =  1.9599640):  0.1507999E+01
```

```
    Normal PPF of 0.975:                       0.1959964E+01
    Lower 95% (normal) Confidence Limit:       0.8914330E+01
    Upper 95% (normal) Confidence Limit:       0.1193033E+02
    Note: Vangel-Rukhin Maximum Likelihood
          Best Usage: 6 or More Labs


4a. Method: DerSimonian Laird (original variance)
    Estimate of Consensus Mean:                 0.1042238E+02
    Estimate of Variance of Consensus Mean:     0.5142135E+00
    Estimate of Between Lab Variance:           0.1527323E+01
    Standard Uncertainty (k = 1):               0.7170868E+00
    Expanded Uncertainty (k = 2):               0.1434174E+01
    Degrees of Freedom:                                     2
    t Percent Point Value:                      0.4302653E+01
    Lower 95% (t-value) Confidence Limit:       0.7337005E+01
    Upper 95% (t-value) Confidence Limit:       0.1350776E+02
    Note: DerSimonian-Laird Best Usage:
          Any Number of Labs:


4b. Method: DerSimonian Laird - Horn-Horn-Duncan Variance
     Estimate of Consensus Mean:                 0.1042238E+02
     Estimate of Variance of Consensus Mean:     0.8871931E+00
     Estimate of Between Lab Variance:           0.1527323E+01
     Standard Uncertainty (k = 1):               0.9419093E+00
     Expanded Uncertainty (k = 2):               0.1883819E+01
     Degrees of Freedom:                                     2
     t Percent Point Value:                      0.4302653E+01
     Lower 95% (t-value) Confidence Limit:       0.6369672E+01
     Upper 95% (t-value) Confidence Limit:       0.1447509E+02
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


4d. Method: DerSimonian Laird - Bootstrap Variance
     Number of Bootstrap Samples                                   100000
     Estimate of Consensus Mean:                            0.1042238E+02
     Estimate of Variance of Consensus Mean:                0.5139354E+00
     Standard Uncertainty (k = 1):                          0.7168929E+00
     Expanded Uncertainty (k = 2):                          0.1433786E+01
     Lower 95% (percentile bootstrap) Confidence Limit:     0.9018119E+01
     Upper 95% (percentile bootstrap) Confidence Limit:     0.1182797E+02
     Lower 95% (symmetric bootstrap) Confidence Limit:      0.9016790E+01
     Upper 95% (symmetric bootstrap) Confidence Limit:      0.1182797E+02
     K (symmetric bootstrap) Coverage Factor:               0.1960670E+01
     Lower 95% (kernel bootstrap) Confidence Limit:         0.9009351E+01
     Upper 95% (kernel bootstrap) Confidence Limit:         0.1183312E+02
     K (kernel bootstrap) Coverage Factor:                  0.1967856E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


11. Method: BOB (Bound on Bias)
    Estimate of Consensus Mean:           0.1042194E+02
    Within Lab Uncertainty:               0.7146745E-01
    Between Lab Uncertainty:              0.9199114E+00
    Standard Uncertainty (k = 1):         0.9226834E+00
    Expanded Uncertainty (k = 2):         0.1845367E+01
    Lower 95% (k = 2) Confidence Limit:   0.8576578E+01
    Upper 95% (k = 2) Confidence Limit:   0.1226731E+02
    Note: BOB Best Usage:
          5 or Fewer Labs:
```

Table 2: 95% Confidence Limits

Method	Consensus Mean	Lower Limit	Upper Limit	Uncertainty (k*SE)
1. Mandel-Paule	0.1042220E+02	0.8913687E+01	0.1193071E+02	0.1508510E+01
3a. Vangel-Rukhin ML	0.1042233E+02	0.8914330E+01	0.1193033E+02	0.1507999E+01
4a. DerSimonian-Laird (original)	0.1042238E+02	0.7337005E+01	0.1350776E+02	0.3085376E+01
4b. DerSimonian-Laird (H-H-D)	0.1042238E+02	0.6369672E+01	0.1447509E+02	0.4052709E+01
4d. DerSimonian-Laird (perc. bootstrap)	0.1042238E+02	0.9018119E+01	0.1182797E+02	0.1405591E+01
4d. DerSimonian-Laird (symm. bootstrap)	0.1042238E+02	0.9016790E+01	0.1182797E+02	0.1405591E+01
4d. DerSimonian-Laird (kern bootstrap)	0.1042238E+02	0.9009351E+01	0.1183312E+02	0.1413030E+01
11. BOB	0.1042194E+02	0.8576578E+01	0.1226731E+02	0.1845367E+01

Table 3: Standard Uncertainties (k = 1)

Method	Consensus Mean	Standard Uncertainty (k = 1)	Relative Standard Uncertainty (%)
1. Mandel-Paule	0.1042220E+02	0.7696621E+00	0.7384836E+01
3a. Vangel-Rukhin ML	0.1042233E+02	0.7694015E+00	0.7382241E+01
4a. DerSimonian-Laird (original)	0.1042238E+02	0.7170868E+00	0.6880259E+01
4b. DerSimonian-Laird (H-H-D)	0.1042238E+02	0.9419093E+00	0.9037371E+01
4d. DerSimonian-Laird (bootstrap)	0.1042238E+02	0.7168929E+00	0.6878398E+01
11. BOB	0.1042194E+02	0.9226834E+00	0.8853275E+01

Table 4: Expanded Uncertainties (k = 2)

Method	Consensus Mean	Expanded Uncertainty (k = 2)	Relative Expanded Uncertainty (%)
1. Mandel-Paule	0.1042220E+02	0.1539324E+01	0.1476967E+02
3a. Vangel-Rukhin ML	0.1042233E+02	0.1538803E+01	0.1476448E+02
4a. DerSimonian-Laird (original)	0.1042238E+02	0.1434174E+01	0.1376052E+02
4b. DerSimonian-Laird (H-H-D)	0.1042238E+02	0.1883819E+01	0.1807474E+02
4d. DerSimonian-Laird (bootstrap)	0.1042238E+02	0.1433786E+01	0.1375680E+02
11. BOB	0.1042194E+02	0.1845367E+01	0.1770655E+02

7.3.5. SRM 2686b Compound: Periclase

```
            Consensus Means Analysis
               (Full Sample Case)

Data Summary:
Response Variable: Y5
Lab-ID Variable: METHOD
Number of Observations:                               34
Grand Mean:                                   0.3342941E+01
Grand Standard Deviation:                     0.5100061E+00
Total Number of Labs:                                  3
Minimum Lab Mean:                             0.2737000E+01
Maximum Lab Mean:                             0.3945000E+01
Minimum Lab SD:                               0.9586528E-01
Maximum Lab SD:                               0.1622789E+00
Mean of Lab Means:                            0.3309278E+01
SD of Lab Means:                              0.6064939E+00
SD of Lab Means (wrt to grand mean):          0.6078937E+00
Within Lab (pooled) SD:                       0.1203492E+00
Within Lab (pooled) Variance:                 0.1448392E-01

            Table 1: Summary Statistics by Lab
```

Lab ID	n(i)	Mean	Variance	Standard Deviation	Standard Deviation of the Mean
1	12	0.3945000E+01	0.1008182E-01	0.1004083E+00	0.2898537E-01
2	12	0.3245833E+01	0.9190152E-02	0.9586528E-01	0.2767392E-01
3	10	0.2737000E+01	0.2633444E-01	0.1622789E+00	0.5131710E-01

```
 1. Method: Mandel-Paule
    Estimate of (unscaled) Consensus Mean:  0.3310202E+01
    Estimate of (scaled) Consensus Mean:    0.4745053E+00
    Between Lab Variance (unscaled):        0.3661977E+00
    Between Lab SD (unscaled):              0.6051427E+00
    Between Lab Variance (scaled):          0.2509468E+00
    Standard Deviation of Consensus Mean:   0.2857286E+00
    Standard Uncertainty (k = 1):           0.2857286E+00
    Expanded Uncertainty (k = 2):           0.5714572E+00
    Expanded Uncertainty (k =  1.9599640):  0.5600178E+00
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.2750185E+01
    Upper 95% (normal) Confidence Limit:    0.3870220E+01
    Note: Mandel-Paule Best Usage:
          6 or More Labs:


 3. Method: Vangel-Rukhin Maximum Likelihood
    Estimate of (unscaled) Consensus Mean:  0.3310666E+01
    Estimate of (scaled) Consensus Mean:    0.4748890E+00
    Between Lab Variance (unscaled):        0.2433602E+00
    Between Lab SD (unscaled):              0.4933155E+00
    Between Lab Variance (scaled):          0.1667691E+00
    Standard Deviation of Consensus Mean:   0.2856400E+00
    Standard Uncertainty (k = 1):           0.2856400E+00
    Expanded Uncertainty (k = 2):           0.5712800E+00
    Expanded Uncertainty (k =  1.9599640):  0.5598441E+00
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.2750822E+01
```

```
    Upper 95% (normal) Confidence Limit:    0.3870510E+01
    Note: Vangel-Rukhin Maximum Likelihood
          Best Usage: 6 or More Labs


4a. Method: DerSimonian Laird (original variance)
    Estimate of Consensus Mean:              0.3311084E+01
    Estimate of Variance of Consensus Mean:  0.6261973E-01
    Estimate of Between Lab Variance:        0.1864500E+00
    Standard Uncertainty (k = 1):            0.2502394E+00
    Expanded Uncertainty (k = 2):            0.5004787E+00
    Degrees of Freedom:                                  2
    t Percent Point Value:                   0.4302653E+01
    Lower 95% (t-value) Confidence Limit:    0.2234391E+01
    Upper 95% (t-value) Confidence Limit:    0.4387777E+01
    Note: DerSimonian-Laird Best Usage:
          Any Number of Labs:


4b. Method: DerSimonian Laird - Horn-Horn-Duncan Variance
     Estimate of Consensus Mean:              0.3311084E+01
     Estimate of Variance of Consensus Mean:  0.1222455E+00
     Estimate of Between Lab Variance:        0.1864500E+00
     Standard Uncertainty (k = 1):            0.3496363E+00
     Expanded Uncertainty (k = 2):            0.6992726E+00
     Degrees of Freedom:                                  2
     t Percent Point Value:                   0.4302653E+01
     Lower 95% (t-value) Confidence Limit:    0.1806720E+01
     Upper 95% (t-value) Confidence Limit:    0.4815448E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


4d. Method: DerSimonian Laird - Bootstrap Variance
     Number of Bootstrap Samples                                   100000
     Estimate of Consensus Mean:                            0.3311084E+01
     Estimate of Variance of Consensus Mean:                0.6282528E-01
     Standard Uncertainty (k = 1):                          0.2506497E+00
     Expanded Uncertainty (k = 2):                          0.5012994E+00
     Lower 95% (percentile bootstrap) Confidence Limit:     0.2818046E+01
     Upper 95% (percentile bootstrap) Confidence Limit:     0.3804407E+01
     Lower 95% (symmetric bootstrap) Confidence Limit:      0.2817761E+01
     Upper 95% (symmetric bootstrap) Confidence Limit:      0.3804407E+01
     K (symmetric bootstrap) Coverage Factor:               0.1968177E+01
     Lower 95% (kernel bootstrap) Confidence Limit:         0.2815512E+01
     Upper 95% (kernel bootstrap) Confidence Limit:         0.3805860E+01
     K (kernel bootstrap) Coverage Factor:                  0.1973975E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


11. Method: BOB (Bound on Bias)
    Estimate of Consensus Mean:           0.3309278E+01
    Within Lab Uncertainty:               0.2170367E-01
    Between Lab Uncertainty:              0.3487196E+00
    Standard Uncertainty (k = 1):         0.3493943E+00
    Expanded Uncertainty (k = 2):         0.6987886E+00
    Lower 95% (k = 2) Confidence Limit:   0.2610489E+01
    Upper 95% (k = 2) Confidence Limit:   0.4008066E+01
    Note: BOB Best Usage:
          5 or Fewer Labs:
```

Table 2: 95% Confidence Limits

Method	Consensus Mean	Lower Limit	Upper Limit	Uncertainty (k*SE)
1. Mandel-Paule	0.3310202E+01	0.2750185E+01	0.3870220E+01	0.5600178E+00
3a. Vangel-Rukhin ML	0.3310666E+01	0.2750822E+01	0.3870510E+01	0.5598441E+00
4a. DerSimonian-Laird (original)	0.3311084E+01	0.2234391E+01	0.4387777E+01	0.1076693E+01
4b. DerSimonian-Laird (H-H-D)	0.3311084E+01	0.1806720E+01	0.4815448E+01	0.1504364E+01
4d. DerSimonian-Laird (perc. bootstrap)	0.3311084E+01	0.2818046E+01	0.3804407E+01	0.4933230E+00
4d. DerSimonian-Laird (symm. bootstrap)	0.3311084E+01	0.2817761E+01	0.3804407E+01	0.4933230E+00
4d. DerSimonian-Laird (kern bootstrap)	0.3311084E+01	0.2815512E+01	0.3805860E+01	0.4955716E+00
11. BOB	0.3309278E+01	0.2610489E+01	0.4008066E+01	0.6987886E+00

Table 3: Standard Uncertainties (k = 1)

Method	Consensus Mean	Standard Uncertainty (k = 1)	Relative Standard Uncertainty (%)
1. Mandel-Paule	0.3310202E+01	0.2857286E+00	0.8631756E+01
3a. Vangel-Rukhin ML	0.3310666E+01	0.2856400E+00	0.8627872E+01
4a. DerSimonian-Laird (original)	0.3311084E+01	0.2502394E+00	0.7557626E+01
4b. DerSimonian-Laird (H-H-D)	0.3311084E+01	0.3496363E+00	0.1055957E+02
4d. DerSimonian-Laird (bootstrap)	0.3311084E+01	0.2506497E+00	0.7570020E+01
11. BOB	0.3309278E+01	0.3493943E+00	0.1055802E+02

Table 4: Expanded Uncertainties (k = 2)

Method	Consensus Mean	Expanded Uncertainty (k = 2)	Relative Expanded Uncertainty (%)
1. Mandel-Paule	0.3310202E+01	0.5714572E+00	0.1726351E+02
3a. Vangel-Rukhin ML	0.3310666E+01	0.5712800E+00	0.1725574E+02
4a. DerSimonian-Laird (original)	0.3311084E+01	0.5004787E+00	0.1511525E+02
4b. DerSimonian-Laird (H-H-D)	0.3311084E+01	0.6992726E+00	0.2111914E+02
4d. DerSimonian-Laird (bootstrap)	0.3311084E+01	0.5012994E+00	0.1514004E+02
11. BOB	0.3309278E+01	0.6987886E+00	0.2111605E+02

7.3.6. SRM 2686b Compound: Arcanite

```
         Consensus Means Analysis
               (Full Sample Case)

Data Summary:
Response Variable: Y6
Lab-ID Variable: METHOD
Number of Observations:                              24
Grand Mean:                               0.2045833E+00
Grand Standard Deviation:                 0.1067699E+00
Total Number of Labs:                                 2
Minimum Lab Mean:                         0.1066667E+00
Maximum Lab Mean:                         0.3025000E+00
Minimum Lab SD:                           0.3339388E-01
Maximum Lab SD:                           0.4245318E-01
Mean of Lab Means:                        0.2045833E+00
SD of Lab Means:                          0.1384751E+00
SD of Lab Means (wrt to grand mean):      0.1384751E+00
Within Lab (pooled) SD:                   0.3819309E-01
Within Lab (pooled) Variance:             0.1458712E-02

            Table 1: Summary Statistics by Lab

----------------------------------------------------------------------------
                                                                    Standard
     Lab                                              Standard     Deviation
      ID    n(i)           Mean       Variance       Deviation   of the Mean
----------------------------------------------------------------------------
       1      12  0.1066667E+00  0.1115152E-02  0.3339388E-01  0.9639984E-02
       2      12  0.3025000E+00  0.1802273E-02  0.4245318E-01  0.1225518E-01


 1. Method: Mandel-Paule
    Estimate of (unscaled) Consensus Mean:  0.2044371E+00
    Estimate of (scaled) Consensus Mean:    0.4992535E+00
    Between Lab Variance (unscaled):        0.1905379E-01
    Between Lab SD (unscaled):              0.1380355E+00
    Between Lab Variance (scaled):          0.4968303E+00
    Standard Deviation of Consensus Mean:   0.6923738E-01
    Standard Uncertainty (k = 1):           0.6923738E-01
    Expanded Uncertainty (k = 2):           0.1384748E+00
    Expanded Uncertainty (k =  1.9599640):  0.1357028E+00
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.6873436E-01
    Upper 95% (normal) Confidence Limit:    0.3401399E+00
    Note: Mandel-Paule Best Usage:
          6 or More Labs:


 3. Method: Vangel-Rukhin Maximum Likelihood
    Estimate of (unscaled) Consensus Mean:  0.2042909E+00
    Estimate of (scaled) Consensus Mean:    0.4985069E+00
    Between Lab Variance (unscaled):        0.9466029E-02
    Between Lab SD (unscaled):              0.9729352E-01
    Between Lab Variance (scaled):          0.2468281E+00
    Standard Deviation of Consensus Mean:   0.6923692E-01
    Standard Uncertainty (k = 1):           0.6923692E-01
    Expanded Uncertainty (k = 2):           0.1384738E+00
    Expanded Uncertainty (k =  1.9599640):  0.1357019E+00
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.6858906E-01
```

```
    Upper 95% (normal) Confidence Limit:    0.3399928E+00
    Note: Vangel-Rukhin Maximum Likelihood
          Best Usage: 6 or More Labs


4a. Method: DerSimonian Laird (original variance)
    Estimate of Consensus Mean:              0.2042759E+00
    Estimate of Variance of Consensus Mean:  0.4559948E-02
    Estimate of Between Lab Variance:        0.8998426E-02
    Standard Uncertainty (k = 1):            0.6752738E-01
    Expanded Uncertainty (k = 2):            0.1350548E+00
    Degrees of Freedom:                                  1
    t Percent Point Value:                   0.1270620E+02
    Lower 95% (t-value) Confidence Limit:   -0.6537408E+00
    Upper 95% (t-value) Confidence Limit:    0.1062293E+01
    Note: DerSimonian-Laird Best Usage:
          Any Number of Labs:


4b. Method: DerSimonian Laird - Horn-Horn-Duncan Variance
     Estimate of Consensus Mean:              0.2042759E+00
     Estimate of Variance of Consensus Mean:  0.9587579E-02
     Estimate of Between Lab Variance:        0.8998426E-02
     Standard Uncertainty (k = 1):            0.9791618E-01
     Expanded Uncertainty (k = 2):            0.1958324E+00
     Degrees of Freedom:                                  1
     t Percent Point Value:                   0.1270620E+02
     Lower 95% (t-value) Confidence Limit:   -0.1039867E+01
     Upper 95% (t-value) Confidence Limit:    0.1448419E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


4d. Method: DerSimonian Laird - Bootstrap Variance
     Number of Bootstrap Samples                                     100000
     Estimate of Consensus Mean:                              0.2042759E+00
     Estimate of Variance of Consensus Mean:                  0.4569701E-02
     Standard Uncertainty (k = 1):                            0.6759956E-01
     Expanded Uncertainty (k = 2):                            0.1351991E+00
     Lower 95% (percentile bootstrap) Confidence Limit:  0.7211734E-01
     Upper 95% (percentile bootstrap) Confidence Limit:  0.3367670E+00
     Lower 95% (symmetric bootstrap) Confidence Limit:   0.7178489E-01
     Upper 95% (symmetric bootstrap) Confidence Limit:   0.3367670E+00
     K (symmetric bootstrap) Coverage Factor:            0.1959940E+01
     Lower 95% (kernel bootstrap) Confidence Limit:      0.7122675E-01
     Upper 95% (kernel bootstrap) Confidence Limit:      0.3372295E+00
     K (kernel bootstrap) Coverage Factor:               0.1966781E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


11. Method: BOB (Bound on Bias)
    Estimate of Consensus Mean:           0.2045833E+00
    Within Lab Uncertainty:               0.7796132E-02
    Between Lab Uncertainty:              0.5653221E-01
    Standard Uncertainty (k = 1):         0.5706725E-01
    Expanded Uncertainty (k = 2):         0.1141345E+00
    Lower 95% (k = 2) Confidence Limit:   0.9044884E-01
    Upper 95% (k = 2) Confidence Limit:   0.3187178E+00
    Note: BOB Best Usage:
          5 or Fewer Labs:
```

Table 2: 95% Confidence Limits

Method	Consensus Mean	Lower Limit	Upper Limit	Uncertainty (k*SE)
1. Mandel-Paule	0.2044371E+00	0.6873436E-01	0.3401399E+00	0.1357028E+00
3a. Vangel-Rukhin ML	0.2042909E+00	0.6858906E-01	0.3399928E+00	0.1357019E+00
4a. DerSimonian-Laird (original)	0.2042759E+00	-0.6537408E+00	0.1062293E+01	0.8580168E+00
4b. DerSimonian-Laird (H-H-D)	0.2042759E+00	-0.1039867E+01	0.1448419E+01	0.1244143E+01
4d. DerSimonian-Laird (perc. bootstrap)	0.2042759E+00	0.7211734E-01	0.3367670E+00	0.1324911E+00
4d. DerSimonian-Laird (symm. bootstrap)	0.2042759E+00	0.7178489E-01	0.3367670E+00	0.1324911E+00
4d. DerSimonian-Laird (kern bootstrap)	0.2042759E+00	0.7122675E-01	0.3372295E+00	0.1330492E+00
11. BOB	0.2045833E+00	0.9044884E-01	0.3187178E+00	0.1141345E+00

Table 3: Standard Uncertainties (k = 1)

Method	Consensus Mean	Standard Uncertainty (k = 1)	Relative Standard Uncertainty (%)
1. Mandel-Paule	0.2044371E+00	0.6923738E-01	0.3386732E+02
3a. Vangel-Rukhin ML	0.2042909E+00	0.6923692E-01	0.3389133E+02
4a. DerSimonian-Laird (original)	0.2042759E+00	0.6752738E-01	0.3305694E+02
4b. DerSimonian-Laird (H-H-D)	0.2042759E+00	0.9791618E-01	0.4793329E+02
4d. DerSimonian-Laird (bootstrap)	0.2042759E+00	0.6759956E-01	0.3309228E+02
11. BOB	0.2045833E+00	0.5706725E-01	0.2789438E+02

Table 4: Expanded Uncertainties (k = 2)

Method	Consensus Mean	Expanded Uncertainty (k = 2)	Relative Expanded Uncertainty (%)
1. Mandel-Paule	0.2044371E+00	0.1384748E+00	0.6773465E+02
3a. Vangel-Rukhin ML	0.2042909E+00	0.1384738E+00	0.6778267E+02
4a. DerSimonian-Laird (original)	0.2042759E+00	0.1350548E+00	0.6611389E+02
4b. DerSimonian-Laird (H-H-D)	0.2042759E+00	0.1958324E+00	0.9586658E+02
4d. DerSimonian-Laird (bootstrap)	0.2042759E+00	0.1351991E+00	0.6618455E+02
11. BOB	0.2045833E+00	0.1141345E+00	0.5578876E+02

7.3.7. SRM 2686b Compound: Free Lime

```
            Consensus Means Analysis
               (Full Sample Case)

Data Summary:
Response Variable: Y7
Lab-ID Variable: METHOD
Number of Observations:                              24
Grand Mean:                                0.5312500E+00
Grand Standard Deviation:                  0.1880405E+00
Total Number of Labs:                                 2
Minimum Lab Mean:                          0.5083333E+00
Maximum Lab Mean:                          0.5541667E+00
Minimum Lab SD:                            0.1651904E+00
Maximum Lab SD:                            0.2133055E+00
Mean of Lab Means:                         0.5312500E+00
SD of Lab Means:                           0.3240906E-01
SD of Lab Means (wrt to grand mean):       0.3240906E-01
Within Lab (pooled) SD:                    0.1907710E+00
Within Lab (pooled) Variance:              0.3639356E-01

            Table 1: Summary Statistics by Lab
```

Lab ID	n(i)	Mean	Variance	Standard Deviation	Standard Deviation of the Mean
1	12	0.5541667E+00	0.4549924E-01	0.2133055E+00	0.6157600E-01
2	12	0.5083333E+00	0.2728788E-01	0.1651904E+00	0.4768637E-01

```
 1. Method: Mandel-Paule
    Estimate of (unscaled) Consensus Mean:  0.5255162E+00
    Estimate of (scaled) Consensus Mean:    0.3748998E+00
    Between Lab Variance (unscaled):        0.0000000E+00
    Between Lab SD (unscaled):              0.0000000E+00
    Between Lab Variance (scaled):          0.0000000E+00
    Standard Deviation of Consensus Mean:   0.1519012E-01
    Standard Uncertainty (k = 1):           0.1519012E-01
    Expanded Uncertainty (k = 2):           0.3038025E-01
    Expanded Uncertainty (k =  1.9599640):  0.2977209E-01
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.4957441E+00
    Upper 95% (normal) Confidence Limit:    0.5552883E+00
    Note: Mandel-Paule Best Usage:
          6 or More Labs:

 3. Method: Vangel-Rukhin Maximum Likelihood
    Estimate of (unscaled) Consensus Mean:  0.5254330E+00
    Estimate of (scaled) Consensus Mean:    0.3730841E+00
    Between Lab Variance (unscaled):        0.3658847E-03
    Between Lab SD (unscaled):              0.1912811E-01
    Between Lab Variance (scaled):          0.1741732E+00
    Standard Deviation of Consensus Mean:   0.1516046E-01
    Standard Uncertainty (k = 1):           0.1516046E-01
    Expanded Uncertainty (k = 2):           0.3032093E-01
    Expanded Uncertainty (k =  1.9599640):  0.2971396E-01
    Normal PPF of 0.975:                    0.1959964E+01
    Lower 95% (normal) Confidence Limit:    0.4957191E+00
    Upper 95% (normal) Confidence Limit:    0.5551470E+00
    Note: Vangel-Rukhin Maximum Likelihood
          Best Usage: 6 or More Labs
```

```
4a. Method: DerSimonian Laird (original variance)
    Estimate of Consensus Mean:                 0.5255162E+00
    Estimate of Variance of Consensus Mean:     0.1421471E-02
    Estimate of Between Lab Variance:           0.0000000E+00
    Standard Uncertainty (k = 1):               0.3770241E-01
    Expanded Uncertainty (k = 2):               0.7540481E-01
    Degrees of Freedom:                                     1
    t Percent Point Value:                      0.1270620E+02
    Lower 95% (t-value) Confidence Limit:       0.4646174E-01
    Upper 95% (t-value) Confidence Limit:       0.1004571E+01
    Note: DerSimonian-Laird Best Usage:
          Any Number of Labs:


4b. Method: DerSimonian Laird - Horn-Horn-Duncan Variance
     Estimate of Consensus Mean:                0.5255162E+00
     Estimate of Variance of Consensus Mean:    0.4922976E-03
     Estimate of Between Lab Variance:          0.0000000E+00
     Standard Uncertainty (k = 1):              0.2218778E-01
     Expanded Uncertainty (k = 2):              0.4437556E-01
     Degrees of Freedom:                                    1
     t Percent Point Value:                     0.1270620E+02
     Lower 95% (t-value) Confidence Limit:      0.2435938E+00
     Upper 95% (t-value) Confidence Limit:      0.8074387E+00
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


4d. Method: DerSimonian Laird - Bootstrap Variance
     Number of Bootstrap Samples                              100000
     Estimate of Consensus Mean:                       0.5255162E+00
     Estimate of Variance of Consensus Mean:           0.1497338E-02
     Standard Uncertainty (k = 1):                     0.3869546E-01
     Expanded Uncertainty (k = 2):                     0.7739091E-01
     Lower 95% (percentile bootstrap) Confidence Limit: 0.4501103E+00
     Upper 95% (percentile bootstrap) Confidence Limit: 0.6015492E+00
     Lower 95% (symmetric bootstrap) Confidence Limit:  0.4494833E+00
     Upper 95% (symmetric bootstrap) Confidence Limit:  0.6015492E+00
     K (symmetric bootstrap) Coverage Factor:          0.1964906E+01
     Lower 95% (kernel bootstrap) Confidence Limit:    0.4494129E+00
     Upper 95% (kernel bootstrap) Confidence Limit:    0.6017053E+00
     K (kernel bootstrap) Coverage Factor:             0.1968942E+01
     Note: DerSimonian-Laird Best Usage:
           Any Number of Labs:


11. Method: BOB (Bound on Bias)
    Estimate of Consensus Mean:           0.5312500E+00
    Within Lab Uncertainty:               0.3894096E-01
    Between Lab Uncertainty:              0.1323094E-01
    Standard Uncertainty (k = 1):         0.4112732E-01
    Expanded Uncertainty (k = 2):         0.8225463E-01
    Lower 95% (k = 2) Confidence Limit:   0.4489954E+00
    Upper 95% (k = 2) Confidence Limit:   0.6135046E+00
    Note: BOB Best Usage:
          5 or Fewer Labs:
```

Table 2: 95% Confidence Limits

Method	Consensus Mean	Lower Limit	Upper Limit	Uncertainty (k*SE)
1. Mandel-Paule	0.5255162E+00	0.4957441E+00	0.5552883E+00	0.2977209E-01
3a. Vangel-Rukhin ML	0.5254330E+00	0.4957191E+00	0.5551470E+00	0.2971396E-01
4a. DerSimonian-Laird (original)	0.5255162E+00	0.4646174E-01	0.1004571E+01	0.4790545E+00
4b. DerSimonian-Laird (H-H-D)	0.5255162E+00	0.2435938E+00	0.8074387E+00	0.2819225E+00
4d. DerSimonian-Laird (perc. bootstrap)	0.5255162E+00	0.4501103E+00	0.6015492E+00	0.7603295E-01
4d. DerSimonian-Laird (symm. bootstrap)	0.5255162E+00	0.4494833E+00	0.6015492E+00	0.7603295E-01
4d. DerSimonian-Laird (kern bootstrap)	0.5255162E+00	0.4494129E+00	0.6017053E+00	0.7618911E-01
11. BOB	0.5312500E+00	0.4489954E+00	0.6135046E+00	0.8225463E-01

Table 3: Standard Uncertainties (k = 1)

Method	Consensus Mean	Standard Uncertainty (k = 1)	Relative Standard Uncertainty (%)
1. Mandel-Paule	0.5255162E+00	0.1519012E-01	0.2890515E+01
3a. Vangel-Rukhin ML	0.5254330E+00	0.1516046E-01	0.2885327E+01
4a. DerSimonian-Laird (original)	0.5255162E+00	0.3770241E-01	0.7174356E+01
4b. DerSimonian-Laird (H-H-D)	0.5255162E+00	0.2218778E-01	0.4222092E+01
4d. DerSimonian-Laird (bootstrap)	0.5255162E+00	0.3869546E-01	0.7363323E+01
11. BOB	0.5312500E+00	0.4112732E-01	0.7741613E+01

Table 4: Expanded Uncertainties (k = 2)

Method	Consensus Mean	Expanded Uncertainty (k = 2)	Relative Expanded Uncertainty (%)
1. Mandel-Paule	0.5255162E+00	0.3038025E-01	0.5781029E+01
3a. Vangel-Rukhin ML	0.5254330E+00	0.3032093E-01	0.5770655E+01
4a. DerSimonian-Laird (original)	0.5255162E+00	0.7540481E-01	0.1434871E+02
4b. DerSimonian-Laird (H-H-D)	0.5255162E+00	0.4437556E-01	0.8444185E+01
4d. DerSimonian-Laird (bootstrap)	0.5255162E+00	0.7739091E-01	0.1472665E+02
11. BOB	0.5312500E+00	0.8225463E-01	0.1548323E+02

7.3.8. Lattice Parameters for phases included in the analyses

Alite, C3S, M3
Formula: $Ca_3(SiO_4)O$
Space group No=8 Setting=1
Hermann Mauguin = C1m1 Lattice=Monoclinic Cell Choice=1 Unique Axis=b
a=3.3083 nm b=0.7027 nm c=1.8499 nm β=94.1200°

Belite, β-C_2S
Formula: Ca_2SiO_4
Space group No=14 Setting=7 Hermann Mauguin=P12_1/n1
Lattice=Monoclinic Cell Choice=2 Unique Axis=b
a=0.5512 nm b=0.6757 nm c=0.9313 nm β =94.5810°

Belite, α-C_2S
Formula: Ca_2SiO_4
Space group No=194 Setting=1 Hermann Mauguin=P6 3/m2/m2/c
Lattice=Hexagonal Unique Axis=c
a=0.5420 nm_c=0.7027 nm

Tricalcium Aluminate, orthorhombic
Formula: $Ca_5Al_3NaO_9$
Space group No=61 Setting=1 Hermann Mauguin=P2 1/b2 1/c2 1/a Lattice=Orthorhombic
a=1.0858 nm b=1.0853 nm c=1.5118 nm

Ferrite
Formula: $Ca_2FeAl_2O_5$
Space group No=46 Setting=2 Hermann Mauguin=Ibm2 Lattice=Orthorhombic
a=0.5557 nm b=1.4543 nm c=0.5361 nm

Periclase
Formula: MgO
Space group No=225 Hermann Mauguin=F4/m-32/m

Arcanite
Formula: $K_2(SO_4)$
Space group No=62 Setting=6 Hermann Mauguin=P2 1/n2 1/a2 1/m Lattice=Orthorhombic
a=0.7476 nm b=1.0071 nm c=0.5763 nm

Lime
Formula: CaO
Space group No=225 Hermann Mauguin=F4/m-32/m
a=0.4819 nm

Acknowledgments

The authors gratefully acknowledge the assistance of the Cemex Cement Company, including Hamid Farzam and Anand Krishnan for agreeing to supply the clinker and especially to Jim Martin from the Cemex Victorville plant for his efforts in sampling, delivery, and providing the XRF analyses. The comments and suggestions from reviewers Scott Jones, XXXXXXX are appreciated as well.