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Precision Estimates for AASHTO Test Method T 105, Determined Using CCRL Proficiency Sample Data

#### **DETAILS**

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#### CHAPTER 1. INTRODUCTION AND RESEARCH APPROACH

#### 1.1 INTRODUCTION

Under National Cooperative Highway Research Programs (NCHRP) Project 9-26, the AASHTO Materials Reference Laboratory (AMRL) is conducting a multi-phase research project to improve estimates of precision in AASHTO test methods for various highway construction materials. The report from Phase 1 of Project 9-26 includes precision estimates of selected volumetric properties of HMA using non-absorptive aggregates [1]. The report from Phase 2 discusses the results of an investigation into the cause of variations in HMA bulk specific gravity test results using non-absorptive aggregates [2]. The report from Phase 3 includes a robust technique developed by AMRL for analyzing proficiency sample data for the purpose of obtaining reliable single-operator and multilaboratory estimates of precision [3]. The report from phase 4 includes two parts. Part one covers the precision estimates of selected volumetric properties of HMA using absorptive aggregates. Part two of the report investigates the effect of aging period on the volumetric properties of the absorptive aggregates [4]. The report from Phase 5 includes update of precision estimates for AASHTO Standard Test Method T 269 [5].

This report includes the results for Part 3 of the 3 in Task 1 of NCHRP 9-26 where data from the CCRL Proficiency Sample Program (PSP) collected from the state laboratories are used to update precision estimates for AASHTO Standard Test Method T 105, "Chemical Analysis of Hydraulic Cement" [6].

Data used in this study are from the chemical analysis of hydraulic cement samples that were sent to the state laboratories participating in the CCRL Proficiency Program. The laboratories receive annual or biannual shipments of CCRL paired proficiency samples, which are tested according to specified ASTM test methods [7]. The hydraulic cement samples for the chemical analysis were prepared and tested according to the methods explained in ASTM C 114 [8]. The data from 14 chemical components were analyzed and are provided in this report. The proficiency samples included in these programs cover a wide range of test values and cement types.

The technique developed by AMRL in Phase 3 was utilized for analyzing proficiency sample data. This technique is a four step methodology for shaving off extraneous results and analyzing the core data of a paired data set. The results of the analysis of the "core data" can then be used to obtain reliable single-operator and multilaboratory estimates of precision.

The precision statement for Chemical Analysis of Hydraulic Cement Test Method in this study resulted from analysis of 107 paired data sets from 14 chemical tests on seven shipments of CCRL paired proficiency samples. Only the most recent proficiency samples were used in order to account for changes in test precision resulting from recent improvements in the test methods.

#### 1.1.1 Problem Statement

AASHTO Standard Test Methods applicable to highway materials require periodic studies to determine estimates of precision. Some precision estimates become outdated as a result of improvements in the methods while other estimates need to be verified to see if they are still accurate. Some test methods need to be expanded to take into account a wider range of materials while other newer test methods may not have precision estimates of any kind.

AASHTO T 105 covers the test methods for chemical analysis of hydraulic cements. Although a set of specific chemical test methods are suggested in T 105, any test method of demonstrated acceptable precision and bias may be used for the analysis of hydraulic cements. To ensure the reliability of the user selected test methods, it is important that the precision estimates of T 105 are updated. The precision estimates also need to be expanded to include chemical components that are not included in AASHTO T 105-06 but are frequently measured by in state and private laboratories.

## 1.1.2 Research Objectives

The objective of this study as Part 3 of the 3 in task 1 of NCHRP 9-26A study is to verify, update, and expand single-operator and multilaboratory precision estimates for the AASHTO T 105, "Standard Method of Test for Chemical Analysis of Hydraulic Cement." Only the most recent CCRL proficiency cement samples that were tested according to the latest version of the test method will be analyzed.

#### 1.2 SCOPE OF STUDY

This work is limited to an evaluation of the data collected from the laboratories participating in the chemical analysis of hydraulic cement for the CCRL proficiency sample program. There are a total of 107 data sets analyzed and included in this report. The resulting precision estimates will reflect a wide range of test values and cement types that are included in the scope of the CCRL Proficiency Sample Program.

#### 1.3 PROFICIENCY SAMPLES USED IN STUDY

Included in the study are the most recent CCRL proficiency samples that were tested in accordance to AASHTO T 105-06 (ASTM C 114-05), "Standard Test Method for Chemical Analysis of Hydraulic Cement." The various cement types included in the study are Type I and Type I/II with and without limestone and Type V with limestone. Table 1-1 provides the sample designation and sample type of the CCRL cement samples and the date of the final report on the chemical analysis of the samples.

**Table 1-1- Sample Designation and Date of Final Report of Proficiency Samples** 

Sample Designation	Cement Type	Date of Final Report		
147 & 148	Type I/II	March-03		
149 & 150	Type I	September-03		
151 & 152	Type I	April-04		
153 & 154	Type I	October-04		
155 & 156	Type I	April -05		
157 & 158	Type V w/ limestone (157) & Type I/II w/ limestone (158)	October-05		
159 & 160	Type I w/ limestone (159) & Type I/II w/ limestone (160)	April-06		
161 & 162	Type I (161) & Type I w/ limestone (162)	October-06		
163 & 164	Type I (163) & Type I / II (164)	April-07		
165 & 166	Type I /II w/ limestone	September-07		

## CHAPTER 2. RESULTS OF ANALYSIS AND ESTIMATES OF PRECISION

#### 2.1 TEST DATA

The test data analyzed in this study are the percentages of 14 chemical components of hydraulic cement. Table 2-1 provides the list of components and the number of data sets used for precision estimate determination of each component. To capture the recent advancement in chemical analysis of hydraulic cement, only the most recent sets of CCRL data were analyzed. As indicated from the table, in most cases 8 sets of data were available for each component. The number of data sets that have been analyzed comes to a total of 107. The results of analysis of each data set can be found in Appendices A through N. This chapter includes summaries of the data and the resulting precision estimates.

Table 2-1- Chemical Components and Number of Data Sets Analyzed

Number	Chemical Components	Number of Data Sets Analyzed
1	SiO <sub>2</sub> (silicon dioxide)	8
2	Al <sub>2</sub> O <sub>3</sub> (aluminum oxide)	8
3	Fe <sub>2</sub> O <sub>3</sub> (ferric oxide)	8
4	CaO (calcium oxide)	8
5	MgO (magnesium oxide)	8
6	SO <sub>3</sub> (sulfur trioxide)	8
7	LOI (loss on ignition)	8
8	Na <sub>2</sub> O (sodium oxide)	10
9	K₂O (potassium oxide)	8
10	TiO <sub>2</sub> (titanium dioxide)	8
11	CI (chloride)	8
12	IR (insoluble residue)	8
13	Cx (free calcium oxide)	6
14	CO <sub>2</sub> (carbon dioxide)	3

#### 2.2 ANALYSIS OF THE DATA

Several sets of chemical analysis data of 14 chemical components of hydraulic cement were included in the precision estimate determination for AASHTO T 105. Table 2-2 through Table 2-15 provide the results of the analyses. Precision estimates are based, where appropriate, on either the coefficients of variation (CV%) or the pooled standard deviation (1s) values.

# 2.2.1 Silicon Dioxide (SiO<sub>2</sub>)

There were 8 sets of percent Silicon Dioxide (SiO<sub>2</sub>) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-2 and Appendix A. A review of the data shown in Table 2-2 indicates that there are no specific trends between the averages and standard deviations of SiO<sub>2</sub> measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.119 percent. The corresponding pooled reproducibility sample standard deviation is 0.196 percent. The pooled estimates are derived using the following equation from Ku [9]:

$$s_p = \sqrt{\frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2 + \dots + (n_k - 1)s_k^2}{n_1 + n_2 + \dots + n_k - k}}$$
 (Equation 1)

Where:

 $s_n$  = pooled standard deviation

 $s_k = k^{th}$  standard deviation

 $n_k$  = number of laboratories analyzed resulting in  $k^{th}$  standard deviation

		Average Results			Repeatability			icibility	Reproducibility	
Sample Number	No. of Labs	Odd	Even	1s	Odd Samples	Even Samples	Odd Samples	Odd Samples	Even Samples	Even Samples
		Samples	Samples		CV%	CV%	1s	CV%	1s	CV%
147 & 148	141	19.66	20.24	0.100	0.5	0.5	0.180	0.9	0.140	0.7
149 & 150	154	20.15	20.70	0.090	0.4	0.4	0.200	1.0	0.170	0.8
153 & 154	207	20.87	22.13	0.100	0.5	0.4	0.160	0.8	0.190	0.8
157 & 158	208	21.11	20.84	0.130	0.6	0.6	0.210	1.0	0.220	1.1
159 & 160	209	20.02	20.51	0.100	0.5	0.5	0.210	1.1	0.210	1.0
161 & 162	218	20.38	20.34	0.100	0.5	0.5	0.180	0.9	0.190	0.9
163 & 164	212	20.58	20.20	0.131	0.6	0.6	0.217	1.1	0.182	0.9
165 & 166	221	20.63	19.03	0.164	0.8	0.9	0.201	1.0	0.230	1.2

Table 2-2- Summary of Statistics for % Silicon Dioxide (SiO<sub>2</sub>)

# 2.2.2 Aluminum Oxide (Al<sub>2</sub>O<sub>3</sub>)

There were 8 sets of percent Aluminum Oxide (Al<sub>2</sub>O<sub>3</sub>) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-3 and Appendix B. A review of the data shown in Table 2-3 indicates that there are no specific trends between the averages and standard deviations of Al<sub>2</sub>O<sub>3</sub> measurements; therefore, the form of the precision estimates should be based on the

sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.073 percent. The corresponding pooled reproducibility sample standard deviation is 0.110 percent. The pooled estimates are derived using Equation 1.

	,	Average	Results	Repeatability			Reproducibility		Reproducibility	
Sample Number	No. of Labs	Odd	Even	1s	Odd Samples	Even Samples	Odd Samples	Odd Samples	Even Samples	Even Samples
	Luos	Samples	Samples		CV%	CV%	1s	CV%	1s	CV%
147 & 148	126	4.32	4.79	0.060	1.4	1.3	0.130	2.9	0.130	2.6
149 & 150	144	4.98	4.64	0.050	1.0	1.0	0.160	3.3	0.140	3.1
153 & 154	199	5.02	3.46	0.070	1.3	1.9	0.100	2.1	0.100	3.0
157 & 158	210	3.73	3.99	0.080	2.0	1.9	0.100	2.6	0.130	3.4
159 & 160	196	5.11	5.12	0.050	1.0	1.0	0.100	1.9	0.100	1.9
161 & 162	203	5.18	4.75	0.040	0.9	0.9	0.080	1.6	0.070	1.5
163 & 164	208	4.93	5.13	0.077	1.6	1.5	0.108	2.2	0.092	1.8
165 & 166	218	4.49	5 26	0 114	2.54	22	0.082	1.8	0.141	2.7

Table 2-3- Summary of Statistics for % Aluminum Oxide (Al<sub>2</sub>O<sub>3</sub>)

## 2.2.3 Ferric Oxide (Fe<sub>2</sub>O<sub>3</sub>)

There were 8 sets of percent Ferric Oxide (Fe<sub>2</sub>O<sub>3</sub>) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-4 and Appendix C. A review of the data shown in Table 2-4 indicates that there are no trends between the averages and standard deviations of Fe<sub>2</sub>O<sub>3</sub> measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.029 percent. The corresponding pooled reproducibility sample standard deviation is 0.051 percent. The pooled estimates are derived using Equation 1.

Table 2-4-	Summary	of Statistics	for % Ferr	ic Ovide	$(\mathbf{Fe}_{2}\mathbf{O}_{2})$

		Average Results			Repeatability			Reproducibility		ucibility
Sample Number	No. of Labs	Odd Samples	Even Samples	1s	Odd Samples	Even Samples	Odd Samples	Odd Samples	Even Samples	Even Samples
		Samples	Samples		CV%	CV%	1s	CV%	1s	CV%
147 & 148	143	3.26	3.16	0.027	0.8	0.9	0.051	1.6	0.050	1.6
149 & 150	156	3.03	3.19	0.029	0.9	0.9	0.047	1.6	0.050	1.6
153 & 154	194	3.45	2.91	0.020	0.6	0.7	0.044	1.3	0.039	1.3
157 & 158	211	3.37	3.00	0.022	0.7	0.7	0.065	1.9	0.060	2.0
159 & 160	198	1.99	3.62	0.031	1.6	0.9	0.039	2.0	0.053	1.5
161 & 162	217	3.66	3.53	0.032	0.9	0.9	0.057	1.6	0.055	1.6
163 & 164	208	2.75	4.24	0.037	1.4	0.9	0.041	1.5	0.066	1.5
165 & 166	219	2.90	2.38	0.026	0.89	1.1	0.042	1.5	0.046	1.9

# 2.2.4 Calcium Oxide (CaO)

There were 8 sets of percent Calcium Oxide (CaO) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-5 and Appendix D. A review of the data shown in Table 2-5 indicates that there are no trends between the averages and standard deviations of CaO measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.199 percent. The corresponding pooled reproducibility sample standard deviation is 0.384 percent. The pooled estimates are derived using Equation 1.

Average Results Repeatability Reproducibility Reproducibility No. Sample Odd Even Odd Odd Even Even of Odd Even Number Samples Samples Samples Samples Samples Samples Labs Samples Samples CV% CV% 1s CV% 1s CV% 147 & 148 147 63.88 64.28 0.190 0.3 0.3 0.330 0.5 0.360 0.6 62.23 0.230 149 & 150 153 63.99 0.4 0.4 0.370 0.6 0.350 0.5 153 & 154 208 63.71 63.57 0.170 0.3 0.3 0.330 0.5 0.330 0.5 157 & 158 205 63.49 64.96 0.160 0.3 0.2 0.520 8.0 0.490 8.0 159 & 160 203 64.34 62.90 0.160 0.3 0.3 0.390 0.6 0.390 0.6 161 & 162 0.220 215 63.91 61.88 0.4 0.4 0.370 0.6 0.430 0.7 163 & 164 210 0.212 0.337 0.345 63.98 63.68 0.3 0.3 0.5 0.5 165 & 166 217 62.63 63.47 0.236 0.4 0.4 0.327 0.5 0.390

Table 2-5- Summary of Statistics for % Calcium Oxide (CaO)

## 2.2.5 Magnesium Oxide (MgO)

There were 8 sets of percent Magnesium Oxide (MgO) data included in the precision estimate determination of AASHTO T 105. The results from analyzing of hydraulic cement are found in Table 2-6 and Appendix E. A review of the data shown in Table 2-6 indicates that there are no trends between the averages and standard deviations of MgO measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.049 percent. The corresponding pooled reproducibility sample standard deviation is 0.070 percent. The pooled estimates are derived using Equation 1.

0.6

Average Results Repeatability Reproducibility Reproducibility No. Odd Odd Odd Even Even Sample Even of Odd Even Samples Samples Samples Samples Samples Samples Number 1s Labs Samples Samples CV% CV% CV% CV% 1s1s147 & 148 140 3.50 1.35 0.070 2.0 5.2 0.090 2.7 0.070 5.5 149 & 150 160 2.46 2.21 0.040 1.5 1.7 0.110 4.3 0.090 4.3 153 & 154 198 1.18 2.37 0.040 3.4 1.7 0.050 3.9 0.060 2.4 157 & 158 200 2.53 1.66 0.050 2.0 3.1 0.090 3.5 0.060 3.8 159 & 160 197 1.27 0.92 0.020 2.0 2.7 0.050 4.0 0.060 6.0 161 & 162 206 1.17 3.55 0.060 5.5 1.8 0.050 4.0 0.090 2.4 0.052 163 & 164 203 1.87 1.07 0.038 2.0 3.6 2.8 0.053 4.9

2.23

2.8

0.071

2.7

0.054

2.6

0.058

2.08

Table 2-6- Summary of Statistics for % Magnesium Oxide (MgO)

## 2.2.6 Sulfur Trioxide (SO<sub>3</sub>)

210

2.60

165 & 166

There were 8 sets of percent Sulfur Trioxide (SO<sub>3</sub>) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-7 and Appendix F. A review of the data shown in Table 2-7 indicates that there are no trends between the averages and standard deviations of SO<sub>3</sub> measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.047 percent. The corresponding pooled reproducibility sample standard deviation is 0.076 percent. The pooled estimates are derived using Equation 1.

Table 2-7- Summary of Statistics for % Sulfur Trioxide (SO<sub>3</sub>)

	No.	Average	Results		Repeatability			Reproducibility		ucibility
Sample Number	of Labs	Odd	Even	1s	Odd Samples	Even Samples	Odd Samples	Odd Samples	Even Samples	Even Samples
		Samples	Samples		CV%	CV%	1s	CV%	1s	CV%
147 & 148	153	2.60	2.90	0.050	2.0	1.8	0.070	2.9	0.080	2.8
149 & 150	160	3.48	2.37	0.040	1.3	1.8	0.080	2.3	0.060	2.7
153 & 154	206	2.78	2.73	0.050	1.6	1.7	0.070	2.7	0.070	2.6
157 & 158	200	2.22	2.35	0.030	1.2	1.1	0.070	3.0	0.070	3.1
159 & 160	199	3.68	3.16	0.040	1.2	1.4	0.100	2.6	0.070	2.3
161 & 162	213	2.73	3.00	0.050	1.8	1.7	0.060	2.3	0.080	2.5
163 & 164	211	2.88	3.58	0.051	1.8	1.4	0.070	2.4	0.082	2.3
165 & 166	213	3.23	3.68	0.055	1.69	1.5	0.080	2.5	0.091	2.5

# 2.2.7 Loss on Ignition (LOI)

There were 8 sets of percent Loss on Ignition (LOI) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-8 and Appendix G. A review of the data shown in Table 2-8 indicates that there are no trends between the averages and standard deviations of LOI measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.055 percent. The corresponding pooled reproducibility sample standard deviation is 0.085 percent. The pooled estimates are derived using Equation 1.

Average Results Repeatability Reproducibility Reproducibility No. Odd Odd Odd Even Sample Even Even of Odd Even Samples Samples Samples Samples Samples Samples Number 1sLabs Samples Samples CV% CV% 1sCV% CV% 1s 147 & 148 177 1.61 1.63 0.040 2.2 2.2 0.060 3.9 0.060 3.9 149 & 150 179 1.98 1.66 0.070 3.4 4.1 0.090 4.4 0.080 4.7 0.050 0.080 0.090 153 & 154 199 2.00 1.80 2.7 3.0 4.2 5.0 157 & 158 209 2.91 2.70 0.070 0.110 0.110 2.3 2.5 3.8 4.1 159 & 160 204 2.65 2.48 0.040 1.6 1.7 0.070 2.5 0.070 3.0 161 & 162 213 1.67 2.00 0.060 3.6 3.0 0.080 4.8 0.090 4.4 163 & 164 204 0.040 0.063 0.057 1.43 1.08 2.8 3.7 4.4 5.3 165 & 166 220 2.13 2.39 0.056 2.63 2.4 0.097 4.5 0.110 4.6

Table 2-8- Summary of Statistics for % Loss on Ignition (LOI)

## 2.2.8 Sodium Oxide (Na<sub>2</sub>O)

There were 10 sets of percent sodium oxide (Na<sub>2</sub>O) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-9 and Appendix H. A review of the data shown in Table 2-9 indicates that there are no trends between the averages and standard deviations of Na<sub>2</sub>O measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the ten pairs of samples analyzed is 0.013 percent. The corresponding pooled reproducibility sample standard deviation is 0.024 percent. The pooled estimates are derived using Equation 1.

Table 2-9- Summary of Statistics for % Sodium Oxide (Na<sub>2</sub>O)

		Average Results			Repeatability			Reproducibility		ucibility
Sample Number	No. of Labs	Odd Samples	Even Samples	1s	Odd Samples	Even Samples	Odd Samples	Odd Samples	Even Samples	Even Samples
		~F	~F		CV%	CV%	1s	CV%	1s	CV%
147 & 148	120	0.13	0.13	0.015	11.49	11.6	0.020	15.7	0.018	13.8
149 & 150	132	0.32	0.07	0.022	6.94	33.7	0.031	9.8	0.024	35.6
151 & 152	141	0.27	0.19	0.012	4.53	6.5	0.025	9.2	0.022	11.5
153 & 154	191	0.11	0.11	0.009	7.77	7.8	0.022	19.8	0.024	21.3
155 & 156	200	0.08	0.14	0.012	15.00	8.9	0.025	29.7	0.026	18.9
157 & 158	195	0.21	0.11	0.014	6.65	12.5	0.031	14.3	0.026	22.8
159 & 160	184	0.12	0.06	0.008	7.04	13.6	0.021	17.8	0.021	34.6
161 & 162	193	0.09	0.13	0.012	12.45	9.1	0.023	24.4	0.022	17.5
163 & 164	199	0.20	0.06	0.015	7.78	27.0	0.021	10.9	0.019	34.2
165 & 166	205	0.16	0.16	0.007	4.60	4.7	0.023	14.6	0.024	15.4

# 2.2.9 Potassium Oxide (K<sub>2</sub>O)

There were 8 sets of percent Potassium Oxide (K<sub>2</sub>O) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-10 and Appendix I. A review of the data shown in Table 2-10 indicates that there are no trends between the averages and standard deviations of K<sub>2</sub>O measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.009 percent. The corresponding pooled reproducibility sample standard deviation is 0.016 percent. The pooled estimates are derived using Equation 1.

Table 2-10- Summary of Statistics for % Potassium Oxide (K<sub>2</sub>O)

	_	Average Results		Repeatability			Reprodu	icibility	Reproducibility	
Sample Number	No. of Labs	Odd Samples	Even Samples	1s	Odd Samples	Even Samples	Odd Samples	Odd Samples	Even Samples	Even Samples
		~F	~		CV%	CV%	1s	CV%	1s	CV%
147 & 148	114	0.69	0.82	0.008	1.2	1.0	0.016	2.4	0.017	2.1
149 & 150	128	0.83	0.69	0.009	1.2	1.4	0.019	2.3	0.019	2.7
153 & 154	195	0.42	0.69	0.010	3.1	1.9	0.015	3.5	0.021	3.0
157 & 158	186	0.47	0.31	0.000	1.5	2.2	0.014	3.0	0.012	4.0
159 & 160	188	0.51	0.75	0.007	1.3	0.9	0.012	2.4	0.015	2.0
161 & 162	180	0.63	0.53	0.007	1.1	1.3	0.011	1.7	0.011	2.1
163 & 164	195	0.68	0.38	0.009	1.3	2.3	0.015	2.2	0.013	3.3
165 & 166	197	0.73	1.17	0.014	2.0	1.22	0.017	2.3	0.025	2.1

# 2.2.10 Titanium Dioxide (TiO<sub>2</sub>)

There were 8 sets of percent Titanium Dioxide (TiO<sub>2</sub>) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-11 and Appendix J. A review of the data shown in Table 2-11 indicates that there are no trends between the averages and standard deviations of TiO<sub>2</sub> measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.005 percent. The corresponding pooled reproducibility sample standard deviation is 0.007 percent. The pooled estimates are derived using Equation 1.

Average Results Repeatability Reproducibility Reproducibility No. Odd Odd Odd Sample Even Even Even of Odd Even Number Samples Samples Samples Samples Samples Samples Labs Samples Samples CV% CV% 1sCV% 1sCV% 147 & 148 0.25 0.23 0.003 1.3 0.008 0.008 71 1.4 3.3 3.4 149 & 150 88 0.21 0.30 800.0 3.7 2.6 0.006 3.0 0.009 3.1 153 & 154 139 0.26 0.16 0.006 2.4 3.9 0.008 3.1 0.007 4.1

2.7

2.2

1.2

1.2

1.40

2.1

2.5

1.1

1.1

1.44

0.007

0.009

0.005

0.009

0.006

3.9

3.4

2.0

3.1

2.7

0.004

0.007

0.005

0.010

0.006

1.9

3.1

2.1

2.9

2.9

**Table 2-11- Summary of Statistics for % Titanium Dioxide (TiO<sub>2</sub>)** 

0.22

0.23

0.25

0.33

0.22

0.005

0.006

0.003

0.004

0.003

# 2.2.11 Chloride (CI)

116

143

116

126

143

0.17

0.26

0.23

0.30

0.23

157 & 158

159 & 160

161 & 162

163 & 164

165 & 166

There were 5 sets of percent Chloride (Cl) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-12 and Appendix K. A review of the data shown in Table 2-12 indicates that there are no trends between the averages and standard deviations of Cl measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the five pairs of samples analyzed is 0.002 percent. The corresponding pooled reproducibility sample standard deviation is 0.004 percent. The pooled estimates are derived using Equation 1.

Average Results Reproducibility Reproducibility Repeatability No. Even Odd Odd Odd Even Even Sample of Odd Even Samples Samples Samples Samples Samples Samples Number Labs Samples Samples CV% CV% CV% CV% 1s1s157 & 158 0.009 0.003 0.003 33.39 105.2 0.004 0.003 116.8 66 52.3 159 & 160 76 0.004 0.005 0.002 42.27 28.8 0.003 82.0 0.003 61.1 161 & 162 90 0.017 0.009 0.003 20.29 39.9 0.007 43.9 0.005 58.7 163 & 164 80 0.005 0.004 0.001 24.18 30.6 0.003 48.5 0.003 60.9 165 & 166 82 0.011 0.009 0.001 13.75 16.5 0.005 42.9 0.004 40.6

**Table 2-12- Summary of Statistics for % Chloride (Cl)** 

## 2.2.12 Insoluble Residue (IR)

There were 8 sets of Insoluble Residue (IR) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-13 and Appendix L. A review of the data shown in Table 2-13 indicates that there are no trends between the averages and standard deviations of IR measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.048 percent. The corresponding pooled reproducibility sample standard deviation is 0.080 percent. The pooled estimates are derived using Equation 1.

<b>Table 2-13-</b>	Summary	of Statistics	for %	Incoluble	Residue	(IR)
1 aine 4-13-	Summa v	oi otausuus	101 /0	Insoluble	Nesidue	(IIX)

		Average	Results		Repeatabili	ty	Reprodu	icibility	Reprod	ucibility
Sample Number	No. of Labs	Odd Samples	Even Samples	1s	Odd Samples	Even Samples	Odd Samples	Odd Samples	Even Samples	Even Samples
		F	I		CV%	CV%	1s	CV%	1s	CV%
147 & 148	160	0.20	0.33	0.040	19.1	11.7	0.060	28.4	0.070	20.5
149 & 150	166	0.19	0.17	0.040	20.3	22.4	0.068	35.5	0.071	40.9
153 & 154	175	0.23	0.54	0.060	25.4	11.1	0.080	33.3	0.090	17.0
157 & 158	179	0.50	0.31	0.050	10.0	16.4	0.090	18.5	0.090	28.5
159 & 160	189	0.23	0.23	0.040	19.0	18.9	0.070	32.5	0.080	35.7
161 & 162	197	0.52	0.47	0.051	9.9	10.9	0.096	18.7	0.088	18.8
163 & 164	198	0.23	0.18	0.042	18.4	23.6	0.078	34.2	0.075	42.2
165 & 166	202	0.45	0.16	0.055	12.19	33.7	0.086	19.0	0.075	45.6

## 2.2.13 Free Calcium Oxide

There were 8 sets of percent Free Calcium Oxide data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-14 and Appendix M. A review of the data shown in Table 2-14 indicates that there are no trends between the averages and standard deviations of Free CaO measurements; therefore, form of the precision estimates should be based on the sample standard

deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.125 percent. The corresponding pooled reproducibility sample standard deviation is 0.214 percent. The pooled estimates are derived using Equation 1.

Average Results Repeatability Reproducibility Reproducibility No. Odd Odd Odd Even Even Even Sample of Odd Even Samples Number Samples Samples Samples Samples Samples Labs Samples Samples CV% CV% CV% 1s1sCV% 147 & 148 147 1.22 1.84 0.200 17.4 11.5 0.260 21.2 0.440 24.1 149 & 150 140 0.64 1.45 0.130 20.4 9.0 0.140 22.5 0.240 16.6 153 & 154 160 1.15 0.76 0.100 8.4 12.8 0.220 19.2 0.160 21.5 157 & 158 162 1.12 0.46 0.140 12.2 29.4 0.220 19.7 0.150 31.7 159 & 160 159 1.07 0.080 0.190 1.13 7.6 7.2 0.170 15.7 16.7 161 & 162 167 1.43 0.50 0.150 10.5 30.1 0.250 17.7 0.160 31.4 163 & 164 170 0.64 0.60 0.070 10.9 20.9 0.147 11.6 0.134 24.5 165 & 166 180 0.77 0.97 0.088 11.54 9.08 0.174 22.7 0.217 22.2

Table 2-14- Summary of Statistics for % Free Calcium Oxide (Free CaO)

## 2.2.14 Carbon Dioxide (CO<sub>2</sub>)

There were 3 sets of percent Carbon Dioxide (CO<sub>2</sub>) data included in the precision estimate determination of AASHTO T 105. The results from analyzing the data are found in Table 2-15 and Appendix N. A review of the data shown in Table 2-15 indicates that there are no trends between the averages and standard deviations of CO<sub>2</sub> measurements; therefore, the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for three available pairs of samples analyzed is 0.083 percent. The corresponding pooled reproducibility sample standard deviation is 0.219 percent. The pooled estimates are derived using Equation 1.

Table 2-15- Summary of Statistics for % Carbon Dioxide (CO<sub>2</sub>)

		Average	Results		Repeatabilit	у	Reprodu	ıcibility	Reprod	ucibility
Sample Number	No. of Labs	Odd	Even	1s	Odd Samples	Even Samples	Odd Samples	Odd Samples	Even Samples	Even Samples
	Laus	Samples	Samples	15	CV%	CV%	1s	CV%	1s	CV%
157 & 158	136	1.82	1.93	0.115	6.33	6.0	0.304	16.7	0.223	11.6
159 & 160	141	1.34	1.33	0.064	4.81	4.8	0.175	13.1	0.166	12.5
165 & 166	155	1.48	1.78	0.062	4.16	3.5	0.209	14.1	0.216	12.1

# 2.2.15 Comparison of the Existing and Developed Precision Estimates

Table 2-16 shows the table of precision estimates in AASHTO T 105-06, with the current precisions crossed out and the new precisions underlined. As indicated from the table, the precision estimates of 6 out of 13 chemical components have been changed significantly, which could be due to the changes in chemical analysis procedure. In addition, precision estimate for CO2 has been added to the table since it is being frequently measured in the state and private laboratories.

**Table 2-16- Comparison of the Existing and Proposed Precision Estimates** 

(Column 1) Component	(Column 2) Maximum Difference between <u>Duplicates</u> <sup>d</sup>	(Column 3)  Maximum Difference of the Average of Duplicates from CRM Certificate Values <sup>bcd</sup>	(Column 4) Maximum Difference between Two Laboratories⁵
SiO2 (silicon dioxide)	<del>0.16</del> <u>0.34</u>	± 0.20	<u>0.56</u>
Al₂O₃ (aluminum oxide)	<del>0.20</del> <u>0.21</u>	± 0.20	<u>0.31</u>
Fe2O3 (ferric oxide)	<del>0.10</del> <u>0.08</u>	± 0.10	<u>0.15</u>
CaO (calcium oxide)	<del>0.20</del> <u>0.56</u>	± 0.30	<u>1.09</u>
MgO (magnesium oxide)	<del>0.16</del> _0.14	± 0.20	<u>0.20</u>
SO₃ (sulfur trioxide)	<del>0.10</del> _0.13	± 0.10	<u>0.22</u>
LOI (loss on ignition)	<del>0.10</del> _0.16	± 0.10	<u>0.24</u>
Na <sub>2</sub> O (sodium oxide)	<del>0.03</del> _0.04	± 0.05	<u>0.07</u>
K <sub>2</sub> O (potassium oxide)	<del>0.03</del> _0.03	± 0.05	<u>0.05</u>
TiO2 (titanium dioxide)	<del>0.02</del> _0.01	± 0.03	<u>0.02</u>
P2Os (phosphorus pentoxide)	0.03	± 0.03	
ZnO (zinc oxide)	0.03	± 0.03	
Mn2O3 (manganic oxide)	0.03	± 0.03	
S (sulfide sulfur)	0.01	1	
Cl (chloride)	<del>0.02</del> <u>0.01</u>	ſ	<u>0.01</u>
IR (insoluble residue)	<del>0.10</del> _0.14	7	<u>0.23</u>
Cx (free calcium oxide)	<del>0.20</del> _0.35	ſ	<u>0.61</u>
Alk <sub>ssl</sub> (water-soluble alkali) <sup>e</sup>	0.75/w	ſ	
Chl <sub>sel</sub> (chloroform-soluble organic substances) CO2 (carbon dioxide)	0.004 <u>0.24</u>	ſ	<u>0.62</u>

#### CHAPTER 3. CONCLUSIONS AND RECOMMENDATIONS

#### 3.1 COMMENTARY

This study was conducted to update and expand the precision estimates for AASHTO Standard Test Method T 105, "Chemical Analysis of Hydraulic Cement." The data analyzed in this study are from the most recent data sets collected from the laboratories participating in the CCRL Proficiency Sample Program. The data reflect wide range of test values and cement types. In most cases the data sets used to derive the precision estimate included well over 150 laboratories.

#### 3.2 CONCLUSION

AASHTO T 105 covers the test methods for chemical analysis of hydraulic cements. Although a set of specific chemical test methods are suggested in T 105-03, any test method of demonstrated acceptable precision and bias may be used for analysis of hydraulic cements. Since reliability of the user selected chemical analysis test methods are determined by T 105 precision and bias estimates, the verification and update of the precision estimates are of particular importance. To update and expand T 105 precision estimates, the most recent chemical analysis data that have been collected from the laboratories participating in the CCRL Proficiency Sample Program were analyzed. To capture the advancement in some of the chemical analysis methods only the most recent sets of CCRL data were analyzed. Also, only 14 of the most frequently measured chemical components by the state and private laboratories were included in the analysis. The comparison of the precision estimates developed in this study and the existing precisions in AASHTO T 105-06 indicated significant change in precisions of a number of chemical analysis.

#### 3.3 RECOMMENDATIONS

In order for the precision estimates of AASHTO T 105 to reflect the recent advancement in chemical testing of hydraulic cement, it is recommended that the updated precision and bias statement in Section 3.4 be adopted for AASHTO T 105.

# 3.4 PRECISION STATEMENT FOR AASHTO T 105, "CHEMICAL ANALYSIS OF HYDRAULIC CEMENT"

#### X. Precision and Bias

- **X.1 Precision -** Criteria for judging the acceptability of percentages of chemical components that are obtained using AASHTO T 105 for hydraulic cement are:
- **X.1.1** Single-Operator Precision (Repeatability) The figures in Column 2 of Table X are the standard deviations that have been found to be appropriate for the chemical components in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results exceeds the values given in Table X, Column 3.
- X.1.2 Multilaboratory Precision (Reproducibility) The figures in Column 4 of Table X are the standard deviations that have been found to be appropriate for the chemical components in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results exceeds the values given in Table X, Column 5.

Table X – Precision Estimates

Chemical Components	Standard Deviation (1s) <sup>a</sup>	Acceptable Range of Two Test Results (d2s) <sup>a</sup>	Standard Deviation (1s) <sup>a</sup>	Acceptable Range of Two Test Results (d2s) <sup>a</sup>
	Single Opera	ator Precision:	Multilaborato	ory Precision:
SiO <sub>2</sub> (silicon dioxide)	0.119	0.333	0.196	0.549
Al <sub>2</sub> O <sub>3</sub> (aluminum oxide)	0.073	0.204	0.110	0.308
Fe <sub>2</sub> O <sub>3</sub> (ferric oxide)	0.029	0.081	0.051	0.143
CaO (calcium oxide)	0.199	0.557	0.384	1.075
MgO (magnesium oxide)	0.049	0.137	0.070	0.196
SO <sub>3</sub> (sulfur trioxide)	0.047	0.132	0.076	0.213
LOI (loss on ignition)	0.055	0.154	0.085	0.238
Na <sub>2</sub> O (sodium oxide)	0.013	0.036	0.024	0.067
K <sub>2</sub> O (potassium oxide)	0.009	0.025	0.016	0.045
TiO <sub>2</sub> (titanium dioxide)	0.005	0.014	0.007	0.020
CI (chloride)	0.002	0.006	0.004	0.011
IR (insoluble residue)	0.048	0.134	0.080	0.224
Cx (free calcium oxide)	0.125	0.350	0.214	0.599
CO2 (carbon dioxide)	0.083	0.232	0.219	0.613

<sup>&</sup>lt;sup>a</sup> These values represent the 1s and d2s limits described in ASTM Practice C670.

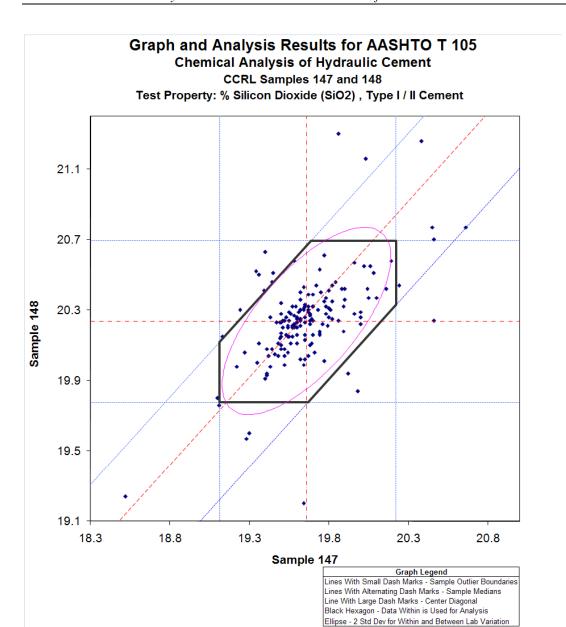
Note – The precision estimates given in Table X are based on the analysis of test results from 107 pairs of CCRL proficiency samples. The data analyzed consisted of results from 66 to 221 laboratories for each of the pairs of samples. The analysis included five cement types: Type I and Type I/II with and without limestone and Type V with limestone.

**X.2 Bias** – No information can be presented on the bias of the procedure because no comparison with the material having an accepted reference value was conducted.

# **REFERENCES**

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- [6] AASHTO, Designation T 105, "Chemical Analysis of Hydraulic Cement" Standard Specifications for Transportation Materials and Methods of Sampling and Testing, 27th Edition, AASHTO, Washington, DC, 2007, CD-ROM.
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- [8] ASTM, Designation D114-06, "Chemical Analysis of Hydraulic Cement," Annual Book of ASTM Standards, Volume 04.01, ASTM, West Conshohocken, PA, 2007.
- [9] Ku, Harry H., "Statistical Concepts in Metrology," NIST Special Publication 300, Volume 1, 1969: p 316-40.

# APPENDIX A: SILICON DIOXIDE (SIO<sub>2</sub>)



Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 147 and 148 Final Report Issued March 2003

Participation: 165 Total Laboratories

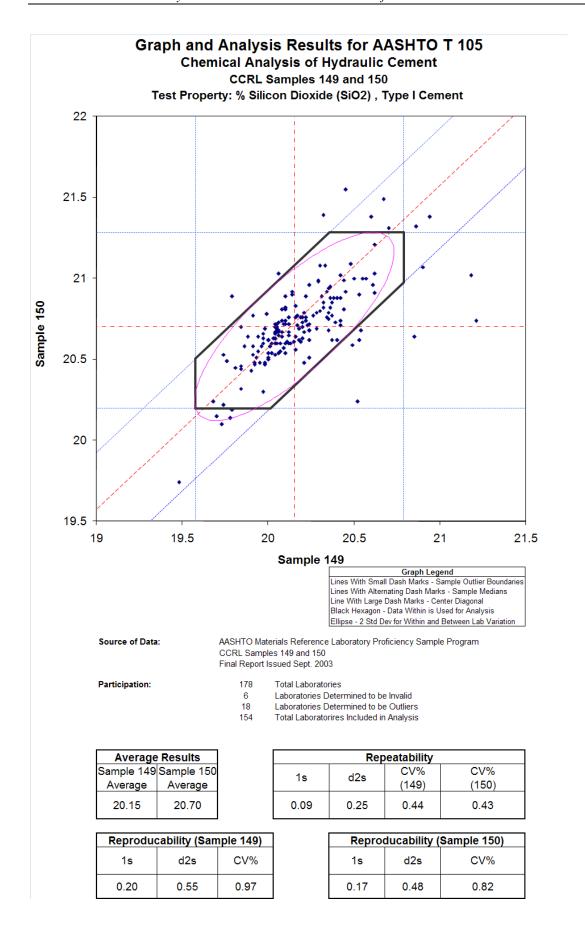
5 Laboratories Determined to be Invalid 19 Laboratories Determined to be Outliers 141 Total Laboratorires Included in Analysis

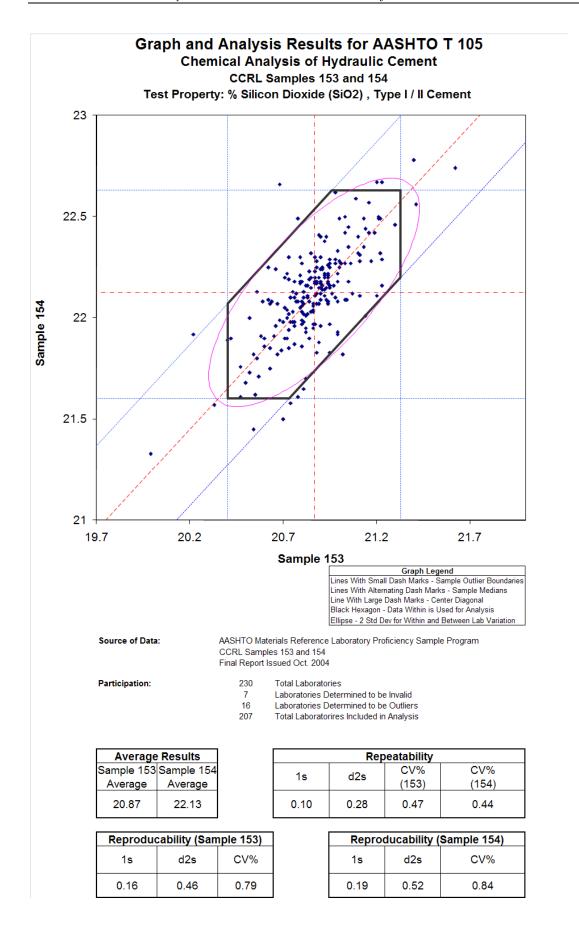
Average Results				
Sample 147	Sample 148			
Average	Average			
19.66	20.24			

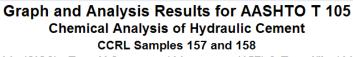
Repeatability					
1s	d2s	CV%	CV%		
15	d2s	(147)	(148)		
0.10	0.28	0.50	0.49		

Reproducability (Sample 147)				
1s	d2s	CV%		
0.18	0.51	0.91		

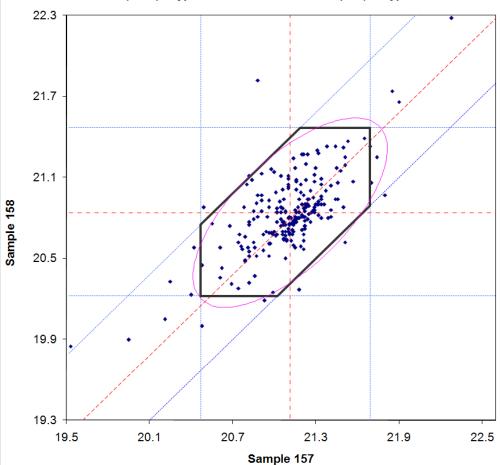
Reproducability (Sample 148)					
1s	d2s	CV%			
0.14	0.41	0.71			







% Silicon Dioxide (SiO2), Type V Cement w/ Limestone (157) & Type I/II w/ Limestone (158)



Graph Legend Lines With Small Dash Marks - Sample Outlier Boundaries Lines With Alternating Dash Marks - Sample Medians Line With Large Dash Marks - Center Diagonal Black Hexagon - Data Within is Used for Analysis Ellipse - 2 Std Dev for Within and Between Lab Variation

AASHTO Materials Reference Laboratory Proficiency Sample Program Source of Data:

CCRL Samples 157 and 158 Final Report Issued Oct. 2005

Participation: 231 Total Laboratories

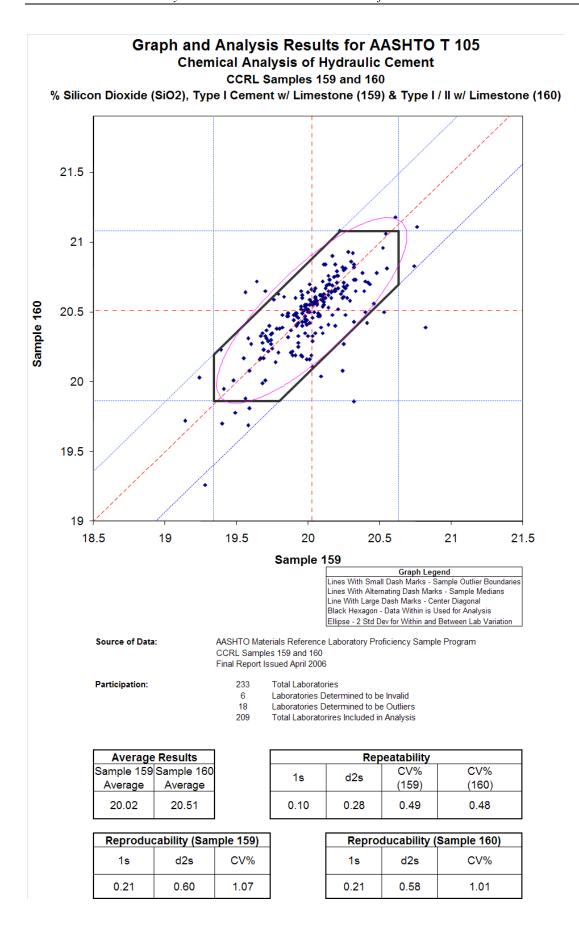
Laboratories Determined to be Invalid Laboratories Determined to be Outliers Total Laboratorires Included in Analysis

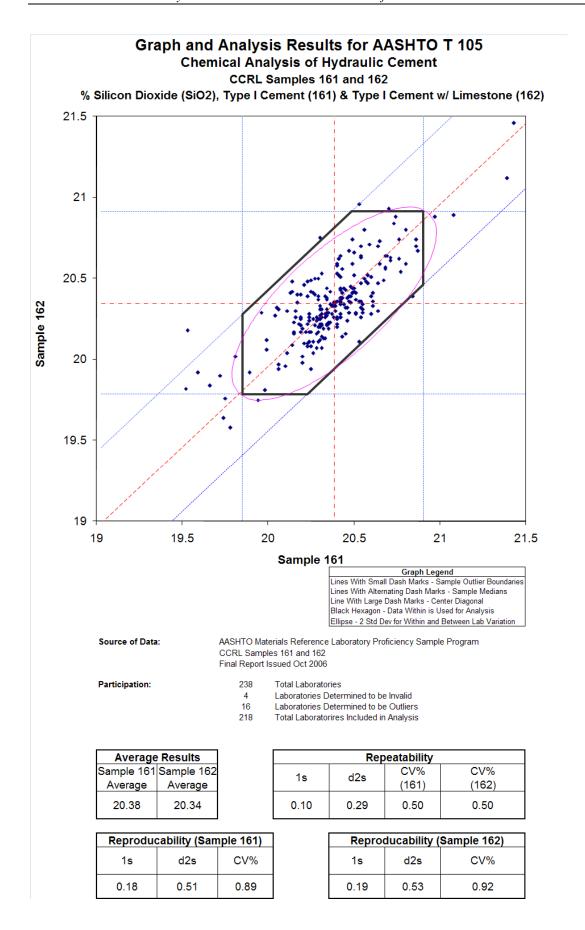
	Results
Sample 157	Sample 158
Average	Average
21.11	20.84

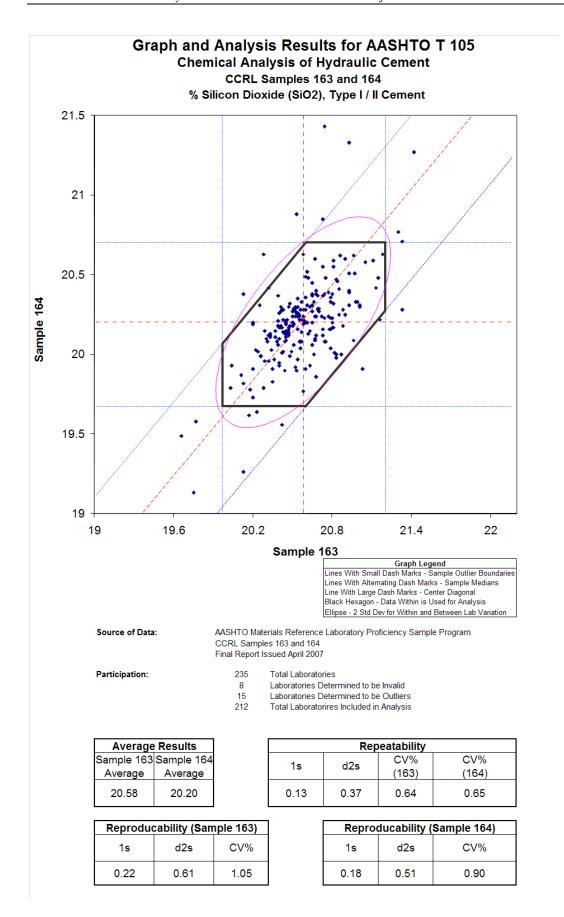
Repeatability					
1s	d2s	CV%	CV%		
15	uzs	(157)	(158)		
0.13	0.38	0.63	0.64		

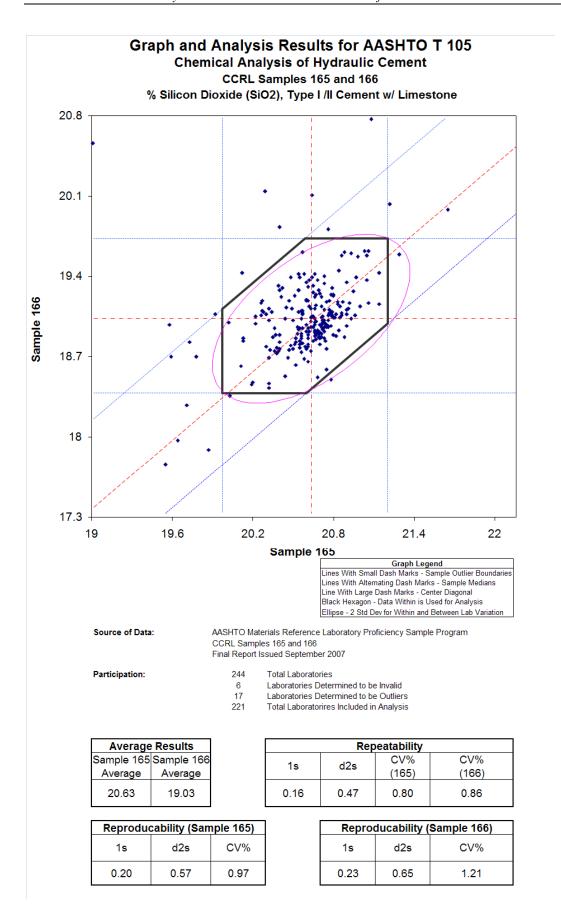
Reproducability (Sample 157)				
1s	d2s	CV%		
0.21	0.59	0.99		

Reproducability (Sample 158)		
1s	d2s	CV%
0.22	0.62	1.05

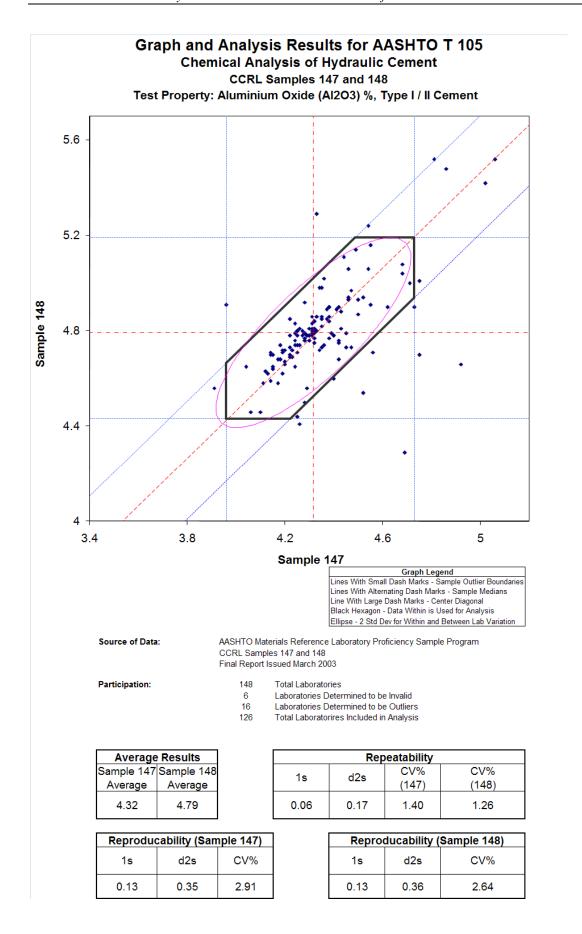


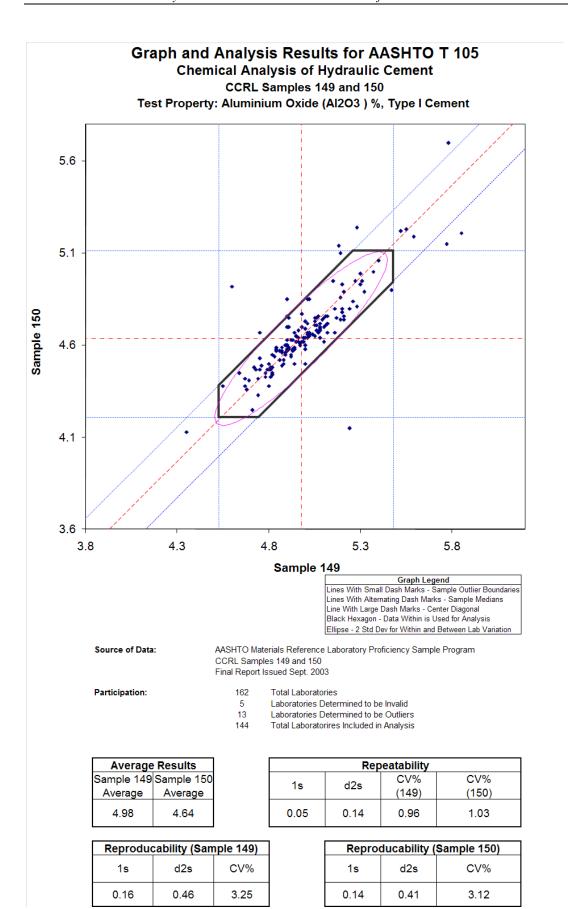


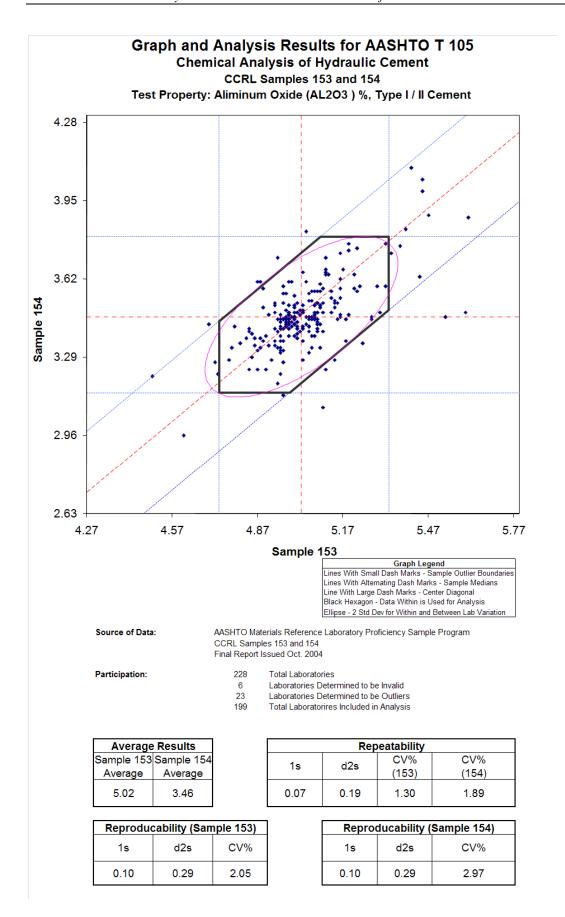




# APPENDIX B: ALUMINUM OXIDE (AL<sub>2</sub>O<sub>3</sub>)

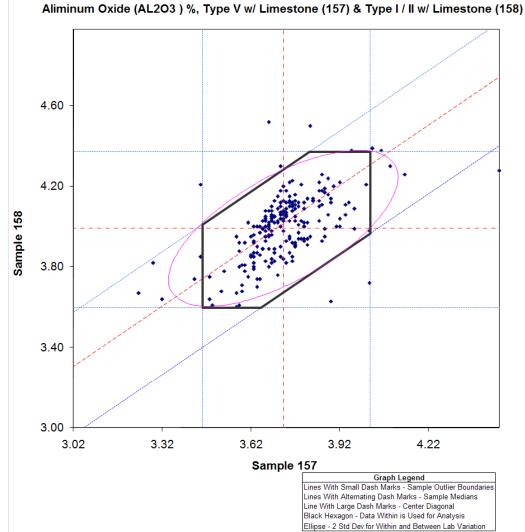






### Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement

CCRL Samples 157 and 158



Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 157 and 158 Final Report Issued Oct. 2005

Participation: 229 Total Laboratories

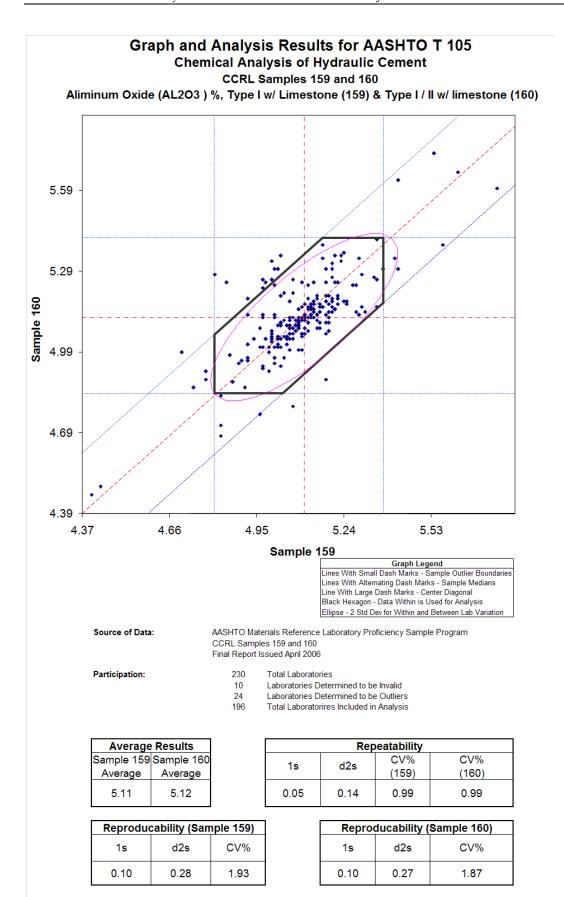
5 Laboratories Determined to be Invalid 14 Laboratories Determined to be Outliers 210 Total Laboratorires Included in Analysis

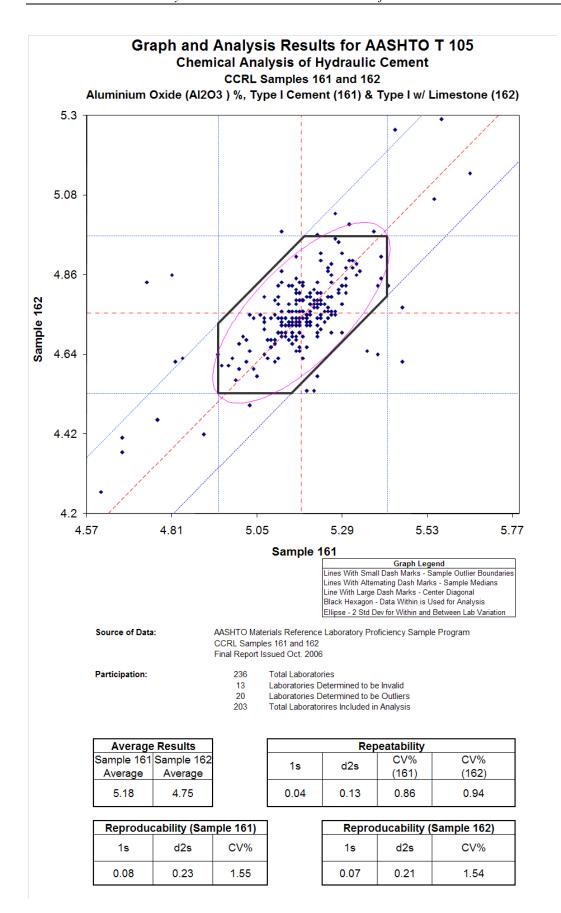
Average Results		
Sample 157 Sample 15		
Average	Average	
3.73	3.99	

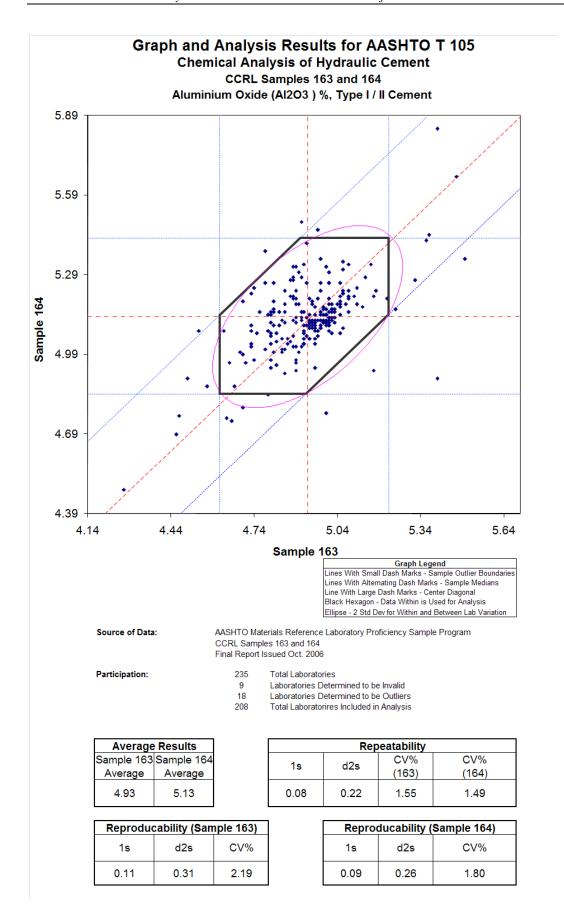
Repeatability			
1s	d2s	CV%	CV%
0	uzs	(157)	(158)
0.08	0.21	2.03	1.89

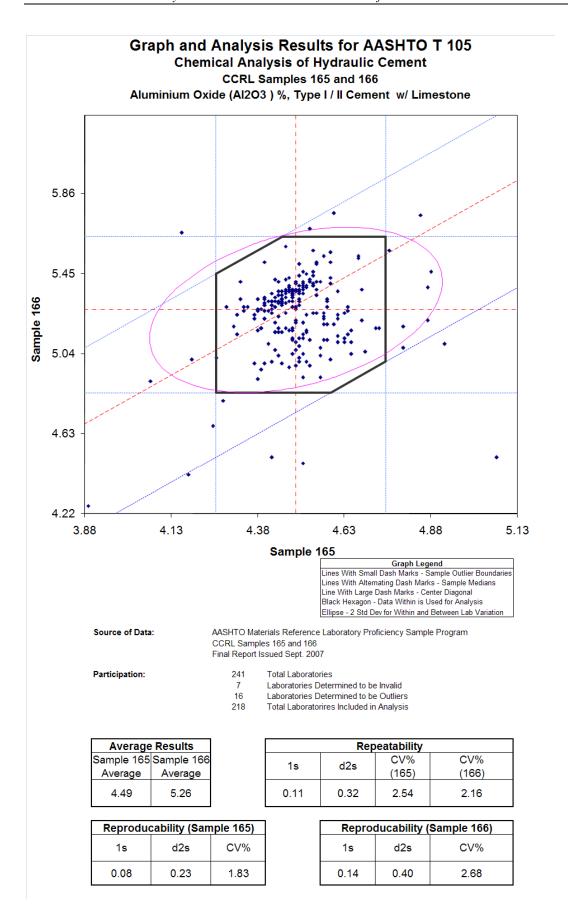
Reproducability (Sample 157)		
1s	d2s	CV%
0.10	0.28	2.64

Reproducability (Sample 158)		
1s d2s		CV%
0.13	0.38	3.37

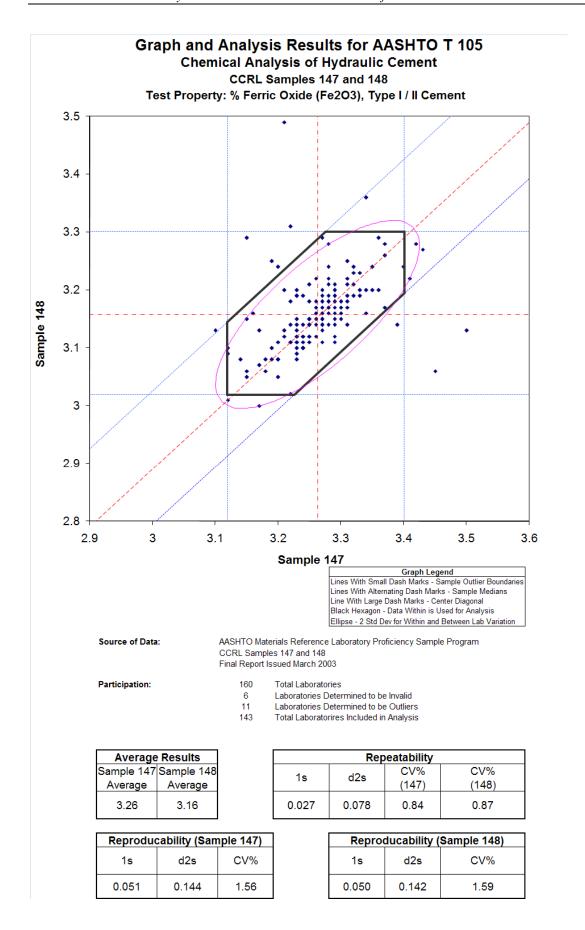


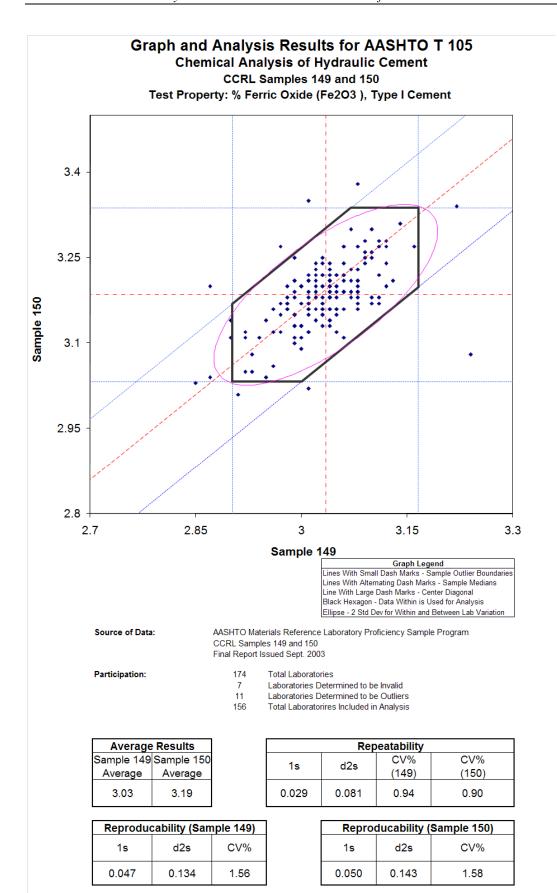


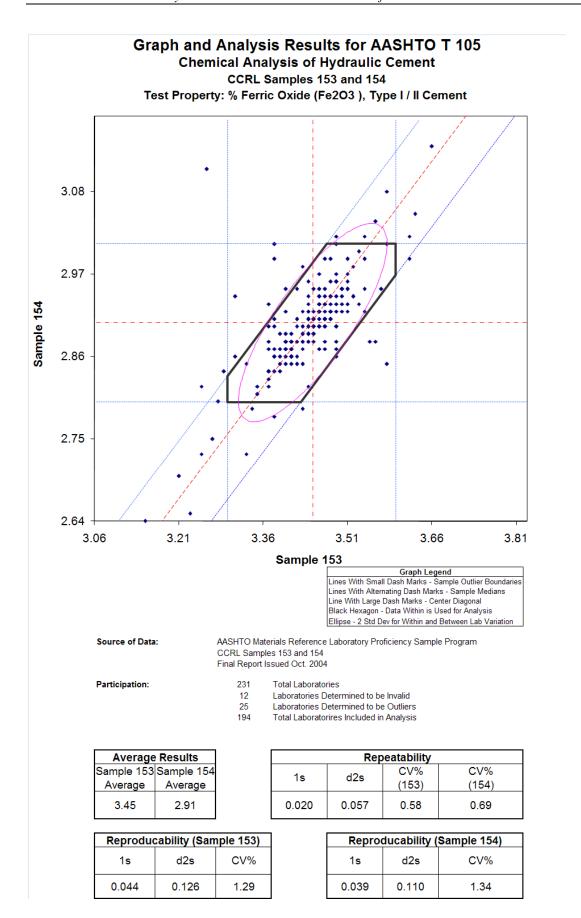


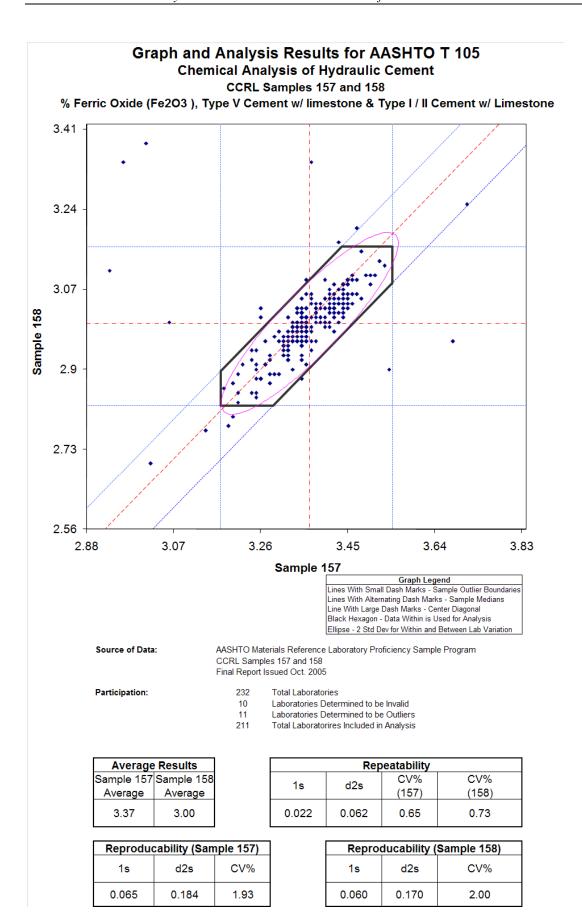


## APPENDIX C: FERRIC OXIDE (FE<sub>2</sub>O<sub>3</sub>)



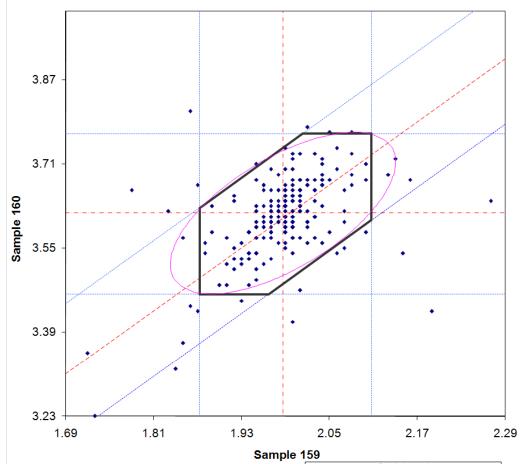






# Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement CCRL Samples 159 and 160 a (Fe2O3). Type I Cement w/ Limestone (159) & Type I / II w/ Li

% Ferric Oxide (Fe2O3 ), Type I Cement w/ Limestone (159) & Type I / II w/ Limestone (160)



Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 159 and 160 Final Report Issued April 2006

Participation: 231 Total Laboratories

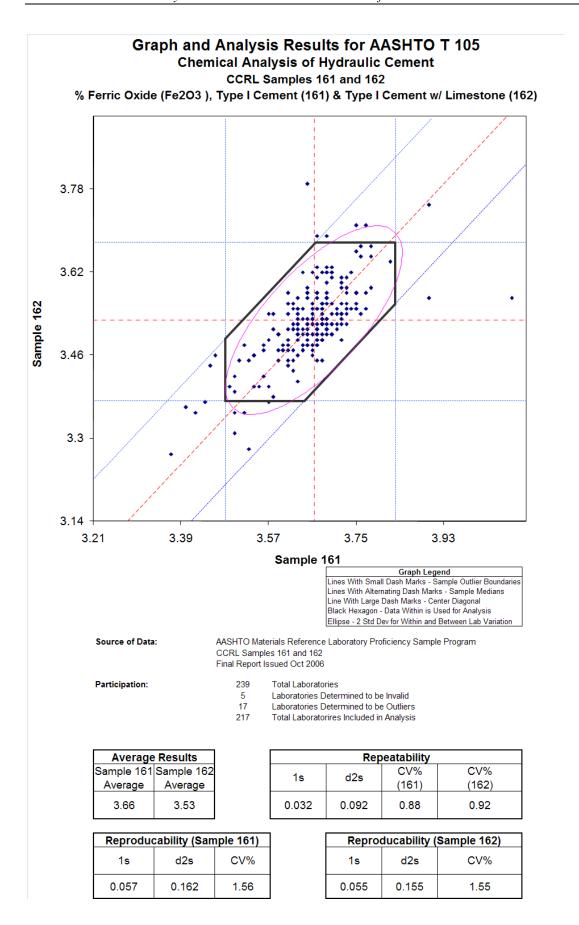
Laboratories Determined to be Invalid
 Laboratories Determined to be Outliers
 Total Laboratorires Included in Analysis

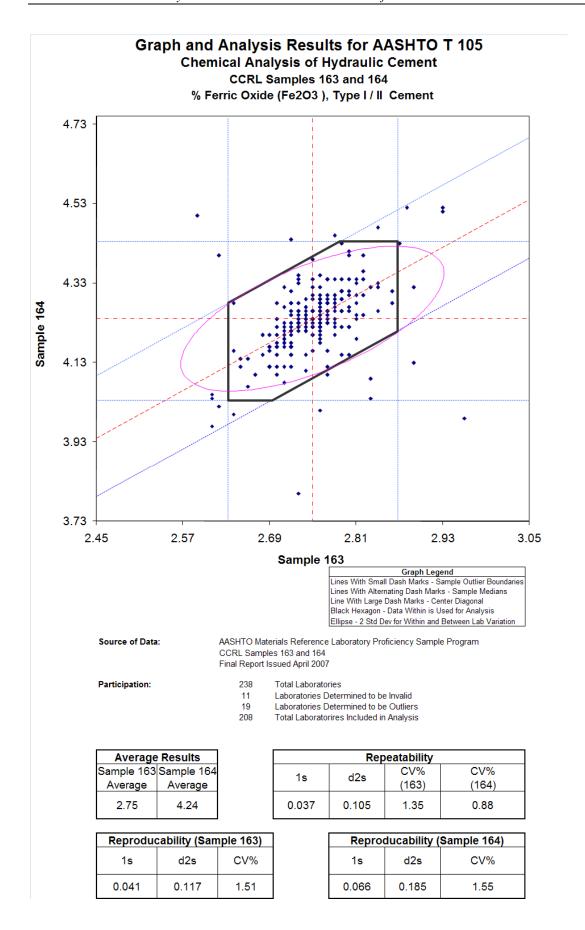
Average Results		
Sample 159 Sample 160		
Average	Average	
1.99	3.62	

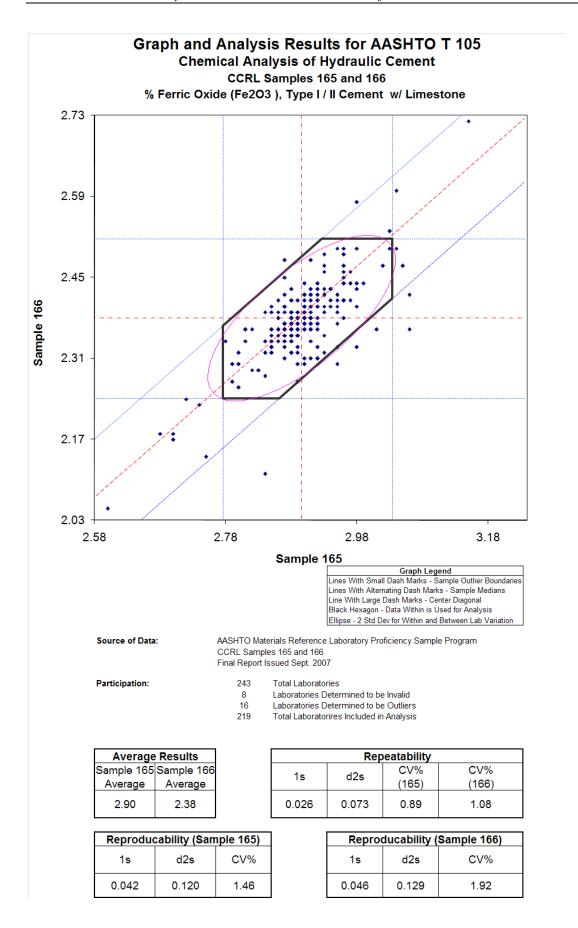
Repeatability			
1s	d2s	CV%	CV%
15	u2s	(159)	(160)
0.031	0.087	1.55	0.85

Reproducability (Sample 159)		
1s	d2s	CV%
0.039	0.111	1.97

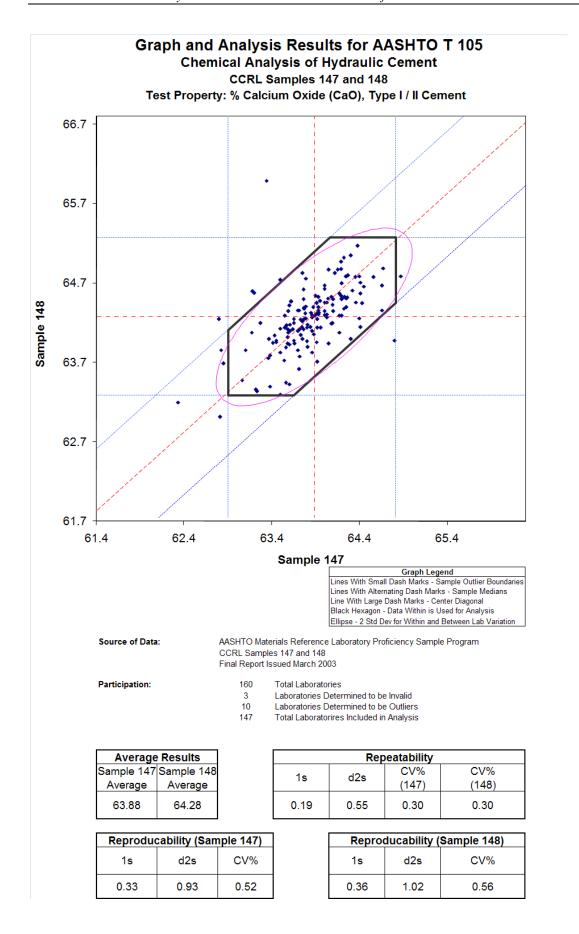
Repro	ducability (	Sample 160)
1s d2s		CV%
0.053	0.149	1.45

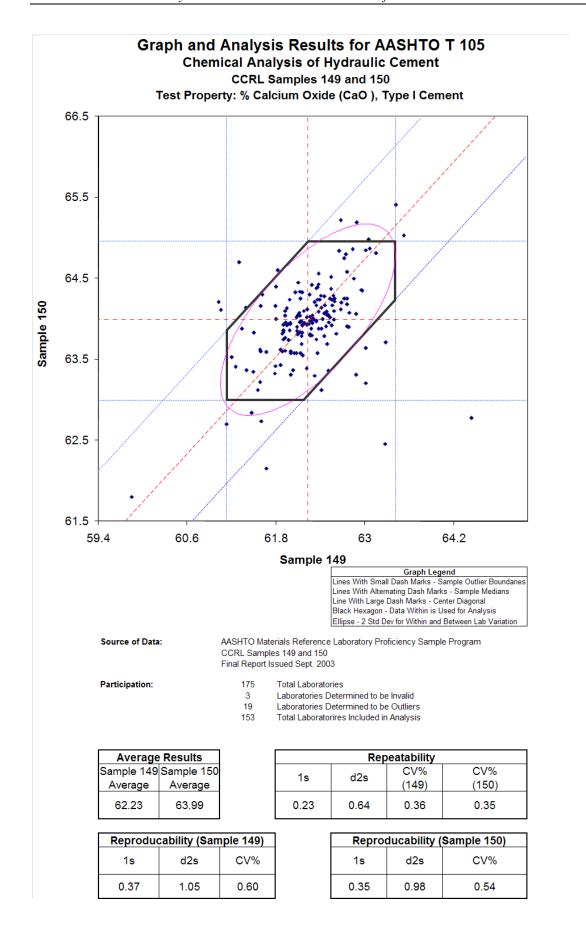


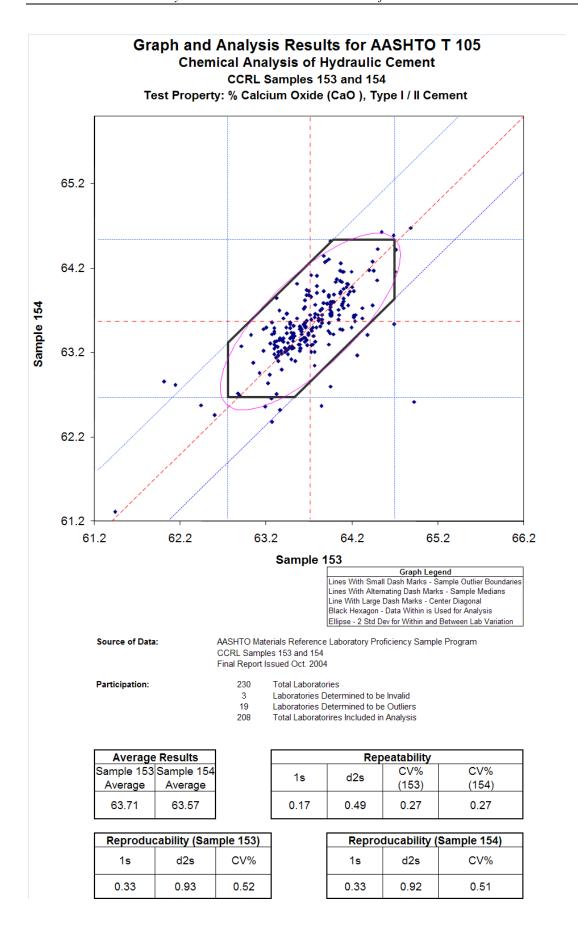




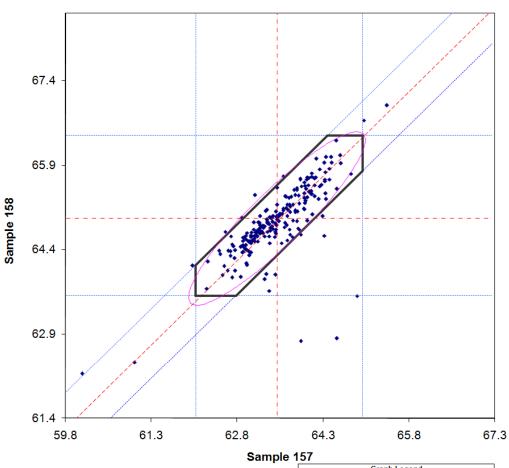
## APPENDIX D: CALCIUM OXIDE (CAO)







# Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement CCRL Samples 157 and 158 % Calcium Oxide (CaO), Type V Cement w/ Limestone (157) & Type I / II w/ Limestone (158)



Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 157 and 158 Final Report Issued Oct. 2005

Participation: 229 Total Laboratories

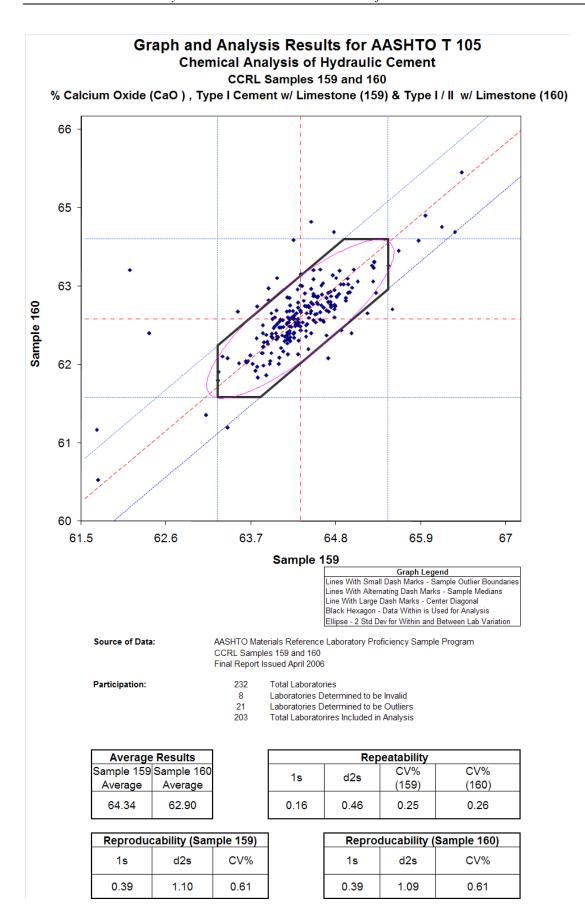
Laboratories Determined to be Invalid
 Laboratories Determined to be Outliers
 Total Laboratorires Included in Analysis

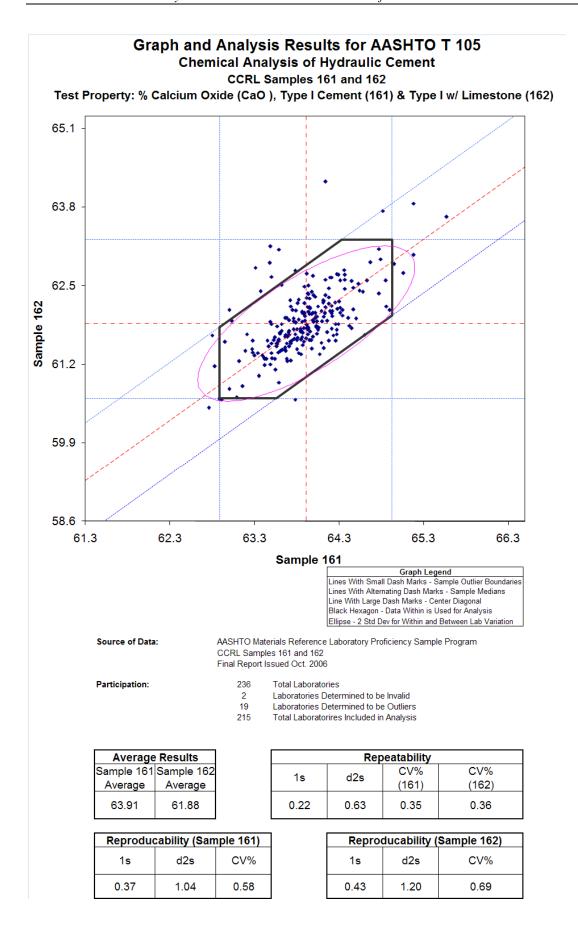
Average Results		
Sample 157	Sample 158	
Average	Average	
63.49	64.96	

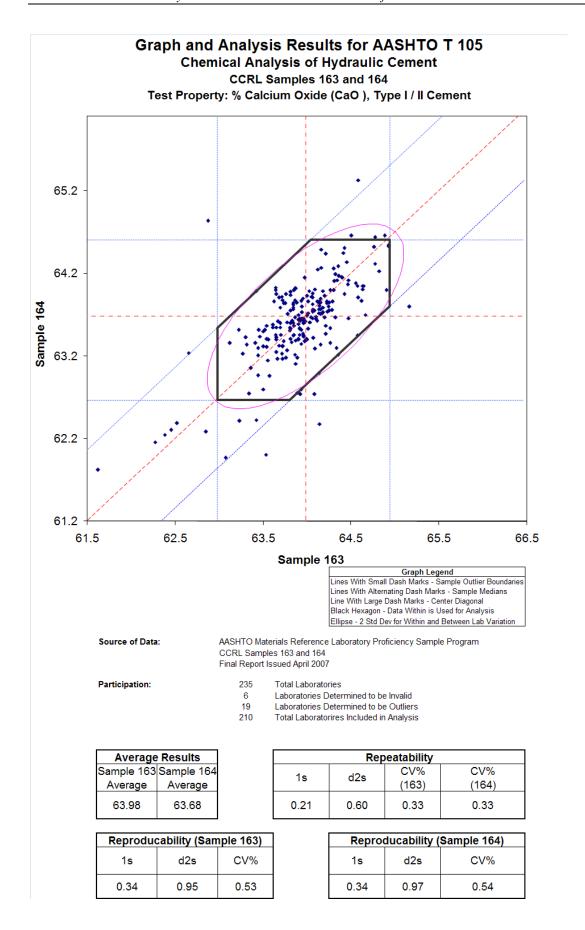
Repeatability			
1s	d2s	CV%	CV%
13	u25	(157)	(158)
0.16	0.44	0.25	0.24

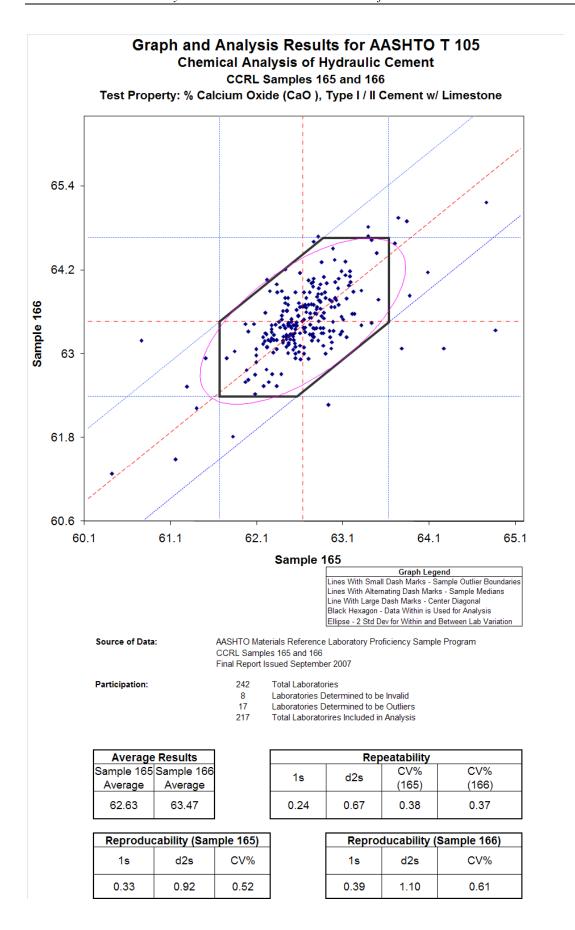
Reproducability (Sample 157)		
1s	d2s	CV%
0.52	1.46	0.82

Repro	ducability (	Sample 158)
1s d2s		CV%
0.49	1.38	0.75

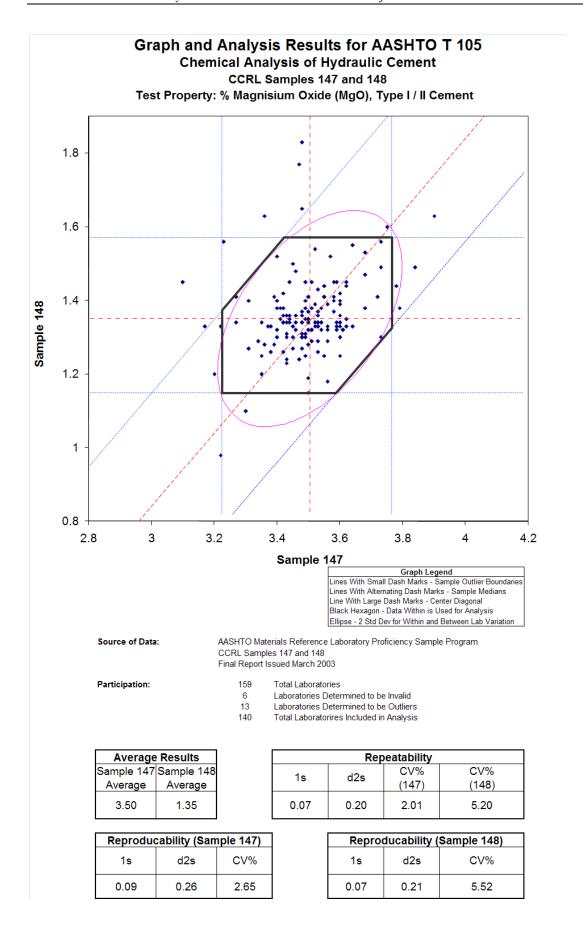


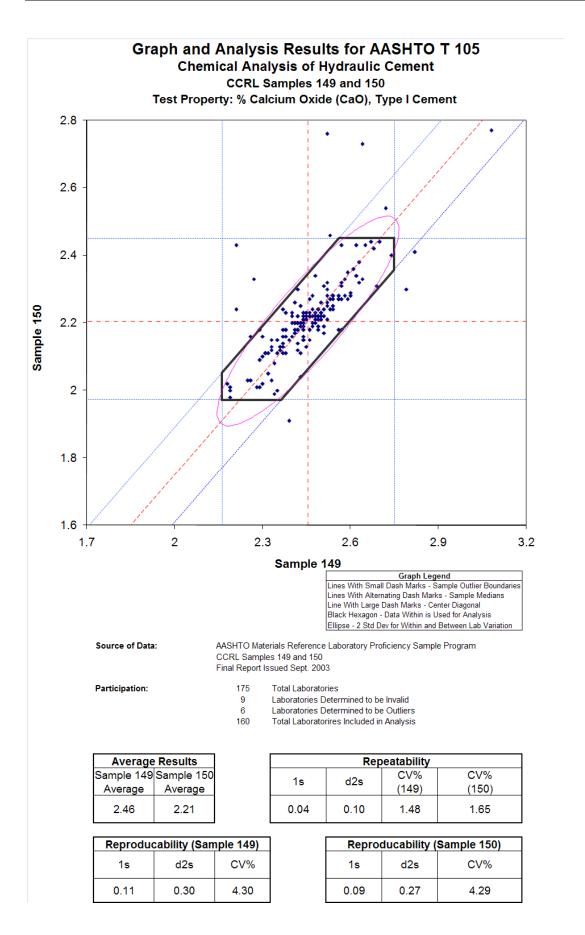


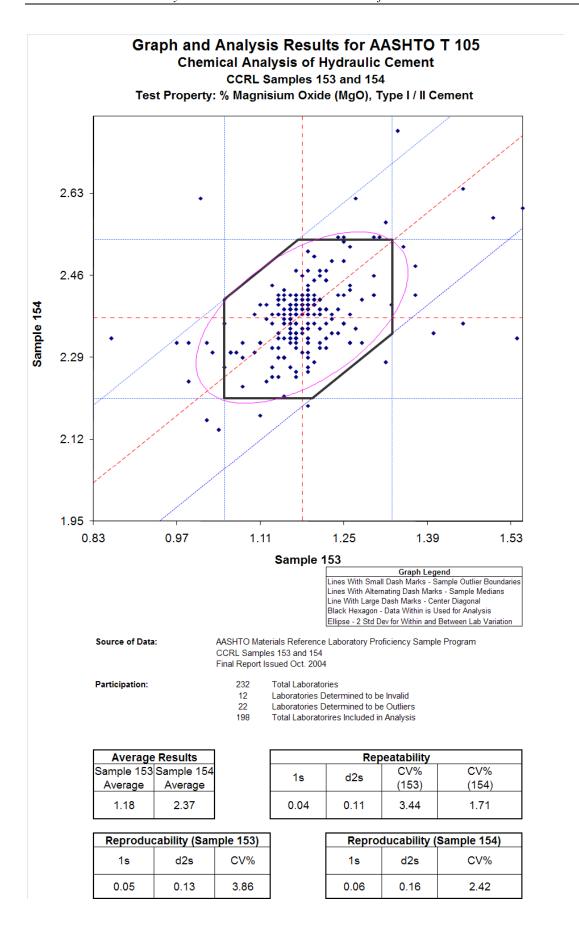


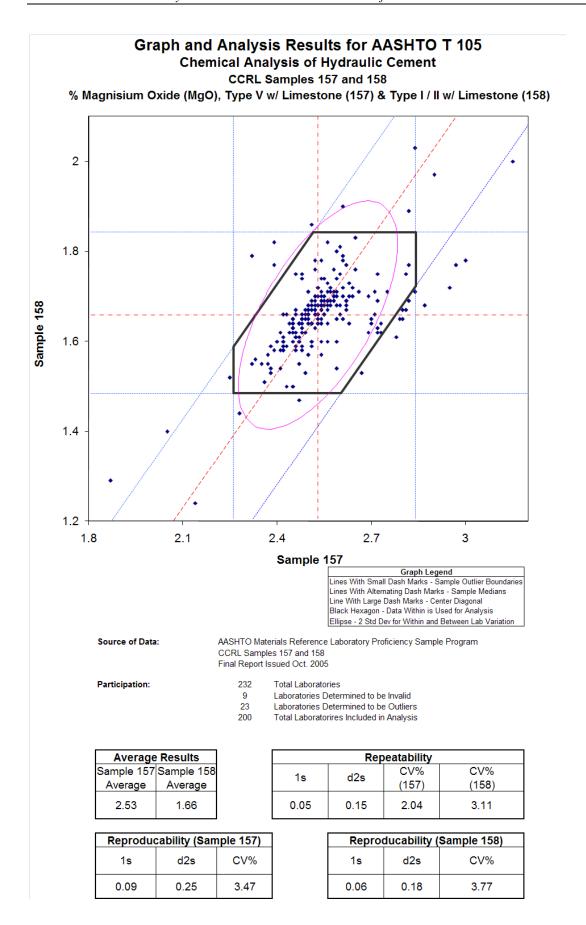


## APPENDIX E: MAGNESIUM OXIDE (MGO)





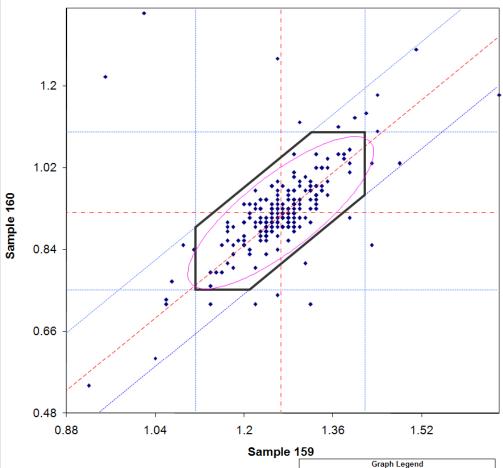




#### Graph and Analysis Results for AASHTO T 105 **Chemical Analysis of Hydraulic Cement**

CCRL Samples 159 and 160

% Magnisium Oxide (MgO), Type I w/ Limestone (159) & Type I / II w/ Limestone (160)



Lines With Small Dash Marks - Sample Outlier Boundaries Lines With Alternating Dash Marks - Sample Medians Line With Large Dash Marks - Center Diagonal Black Hexagon - Data Within is Used for Analysis Ellipse - 2 Std Dev for Within and Between Lab Variatio

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 159 and 160 Final Report Issued April 2006

Participation: 232 Total Laboratories

14 Laboratories Determined to be Invalid

21 Laboratories Determined to be Outliers

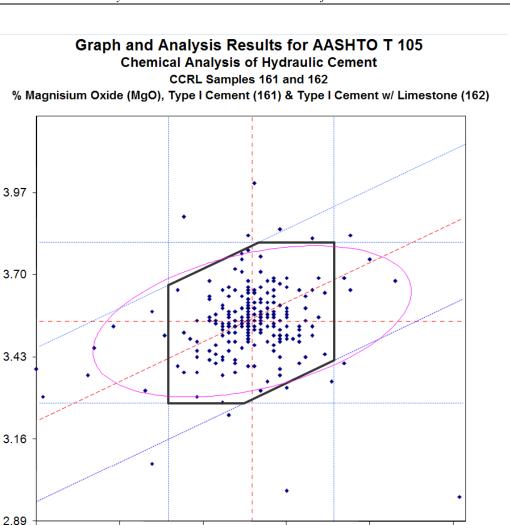
197 Total Laboratorires Included in Analysis

Average Results			
Sample 159	Sample 160		
Average	Average		
1.27	0.92		

	Repeatability			
	1s d2s	CV%	CV%	
		(159)	(160)	
	0.02	0.07	1.96	2.69

Reproducability (Sample 159)			
1s	d2s	CV%	
0.05	0.14	4.00	

Reproducability (		Sample 160)
1s	d2s	CV%
0.06	0.16	5.99



Sample 161

1.22

Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

1.35

1.48

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 161 and 162 Final Report Issued Oct. 2006

1.09

Participation: 237 Total Laboratories

0.96

8 Laboratories Determined to be Invalid 23 Laboratories Determined to be Outliers 206 Total Laboratorires Included in Analysis

Average Results		
Sample 161	Sample 162	
Average	Average	
1.17	3.55	

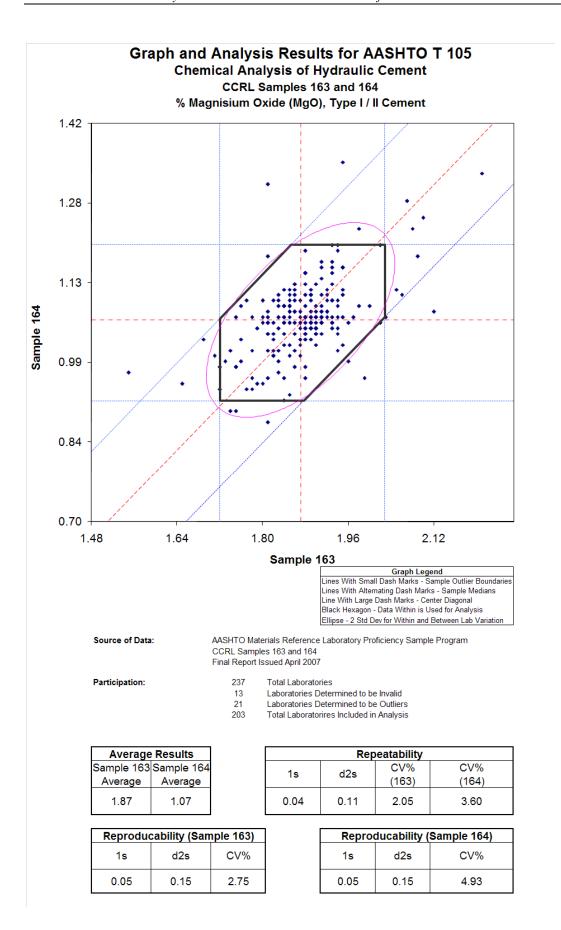
Sample 162

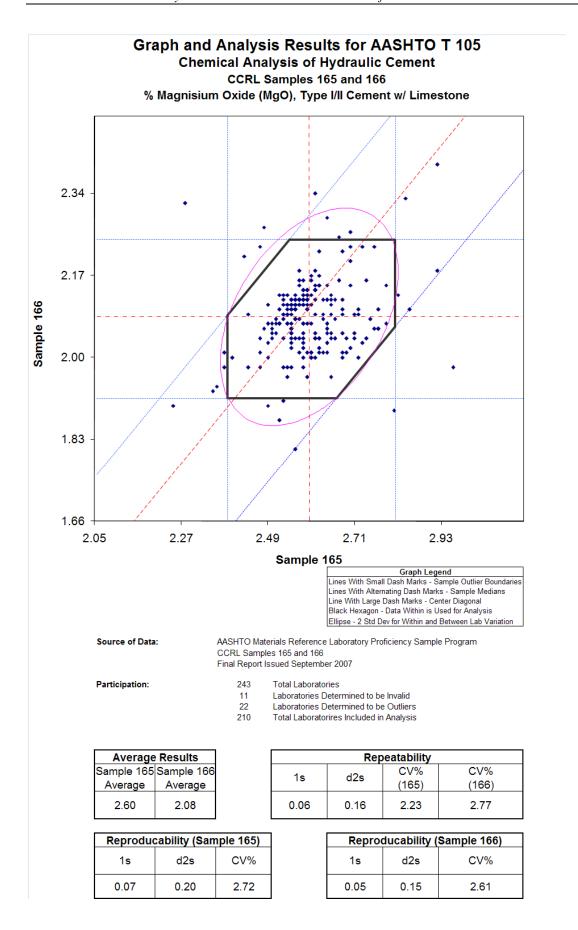
0.83

Repeatability			
10	1s d2s	CV%	CV%
15		(161)	(162)
0.06	0.18	5.49	1.81

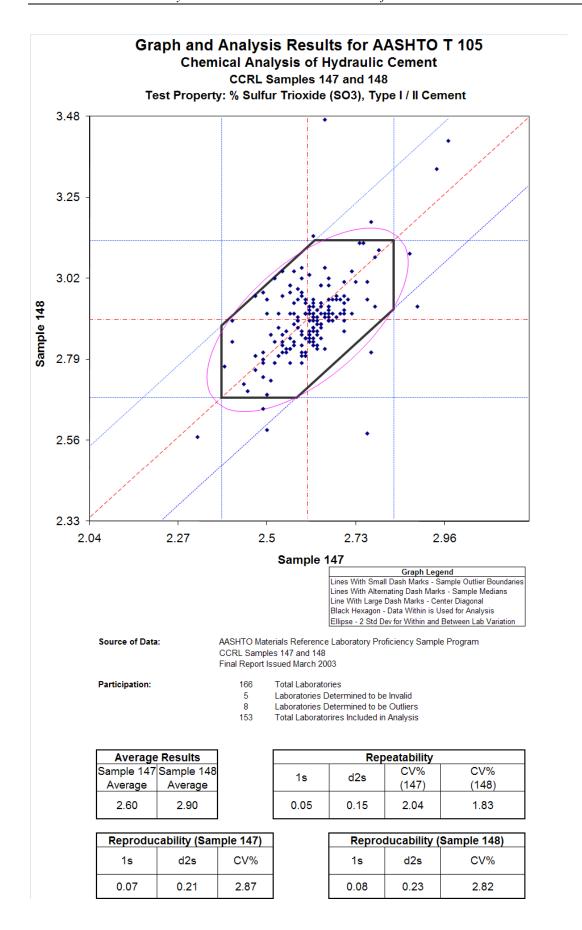
Reproducability (Sample 161)			
1s	d2s	CV%	
0.05	0.13	3.99	

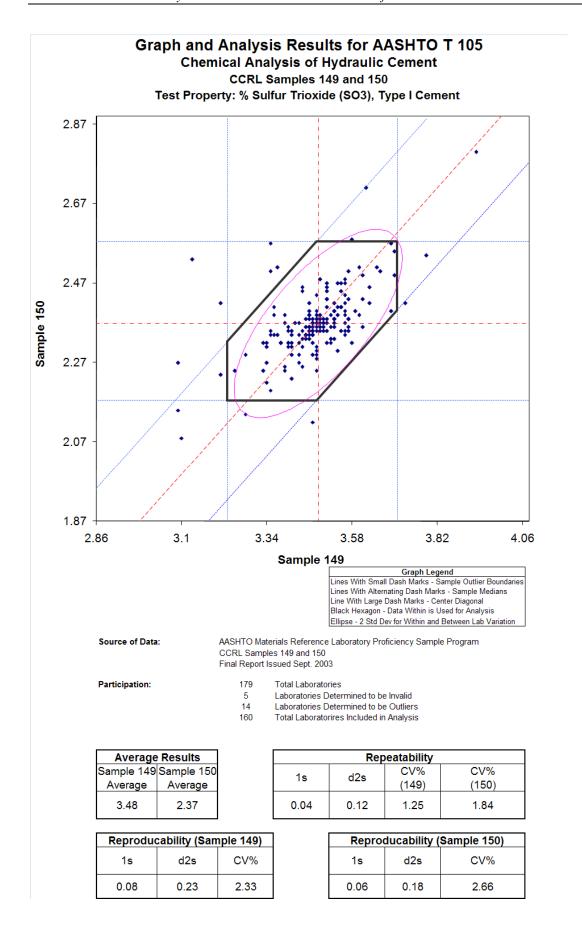
Repro	ducability (	Sample 162)
1s	d2s	CV%
0.09	0.24	2.43

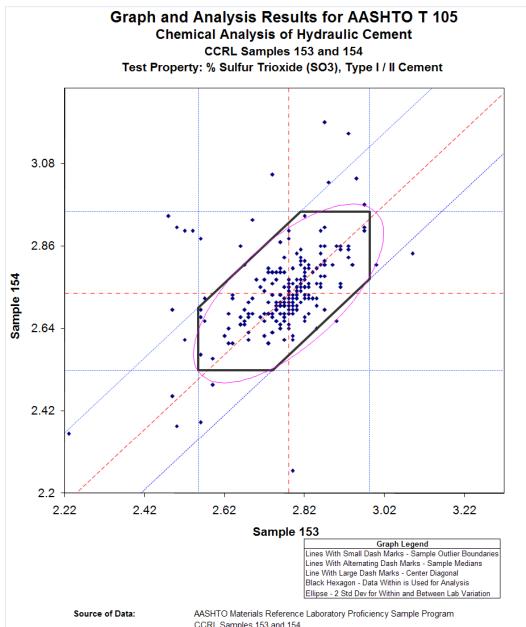




## APPENDIX F: SULFUR TRIOXIDE (SO<sub>3</sub>)







CCRL Samples 153 and 154 Final Report Issued Oct. 2004

Participation: 233 Total Laboratories

10 Laboratories Determined to be Invalid

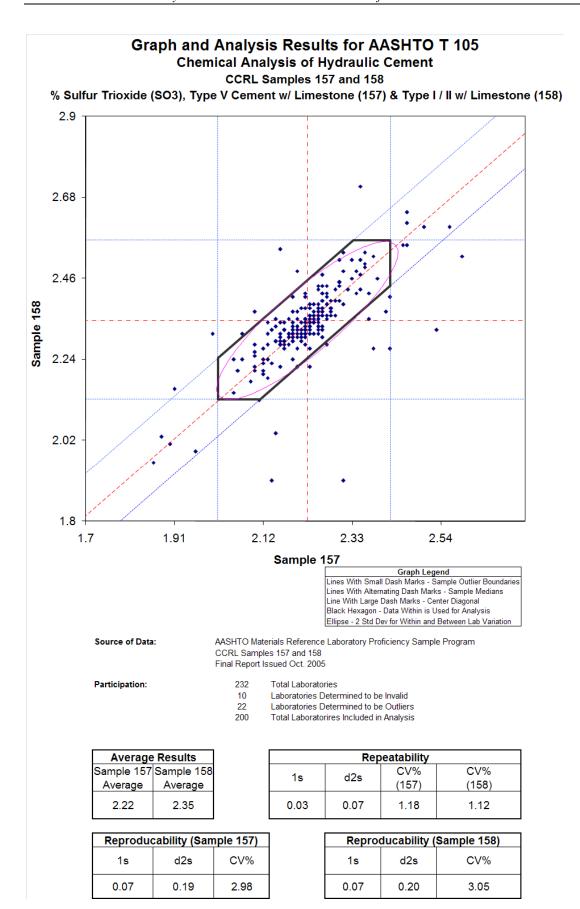
17 Laboratories Determined to be Outliers

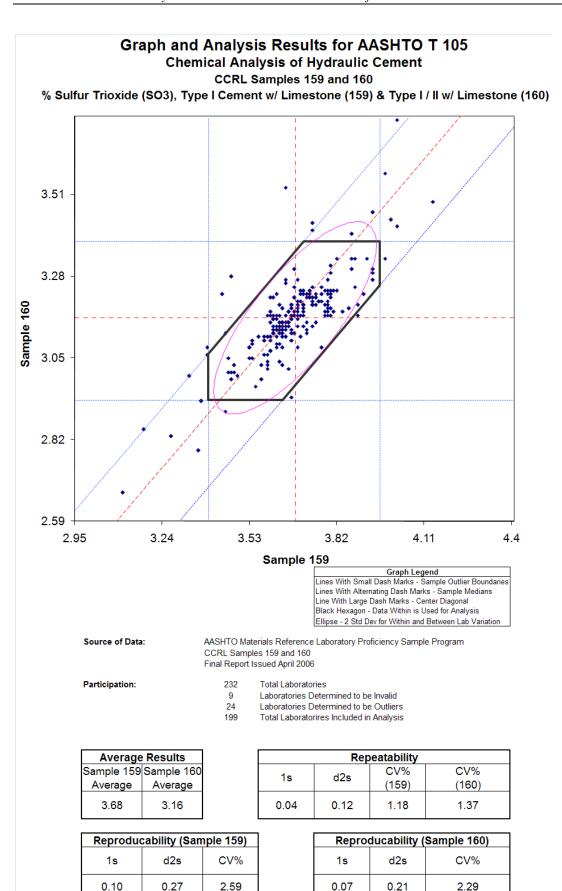
Average Results			
Sample 153 Sample 154			
Average	Average		
2.78	2.73		

Repeatability			
10	1s d2s		CV%
1s	u2s	(153)	(154)
0.05	0.13	1.62	1.65

Reproducability (Sample 153)		
1s	d2s	CV%
0.07	0.21	2.65

Repro	ducability (	Sample 154)
1s	d2s	CV%
0.07	0.20	2.62

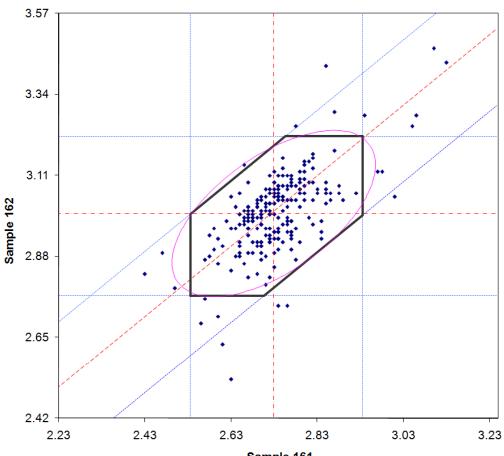




-	O
n	a

# Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement CCRL Samples 161 and 162

Test Property: % Sulfur Trioxide (SO3), Type I Cement (161) & Type I w/ Limestone (162)



**Sample 161** 

Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 161 and 162 Final Report Issued Oct 2006

Participation: 240 Total Laboratories

8 Laboratories Determined to be Invalid

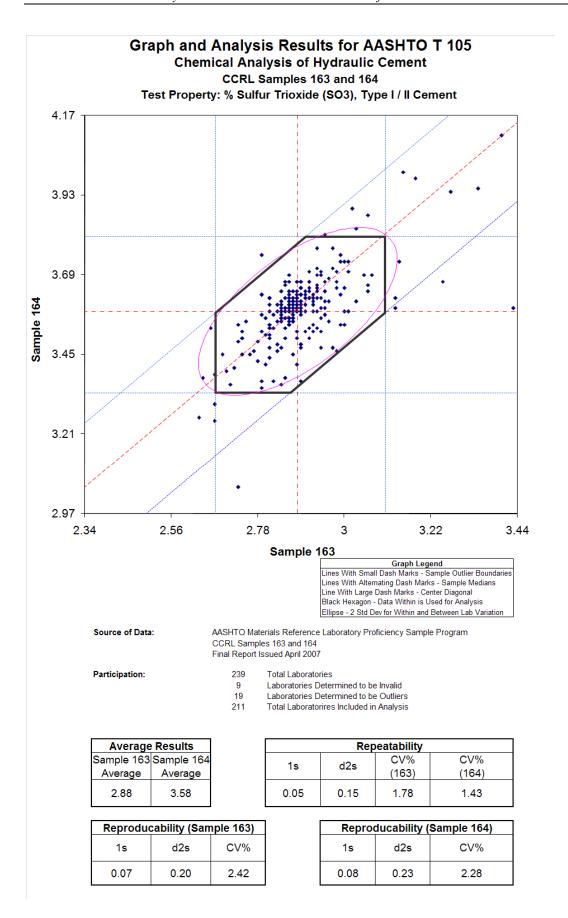
19 Laboratories Determined to be Outliers

Average Results			
Sample 161 Sample 162			
Average	Average		
2.73	3.00		

Repeatability			
1s	1s d2s		CV%
9	3	(161)	(162)
0.05	0.14	1.84	1.67

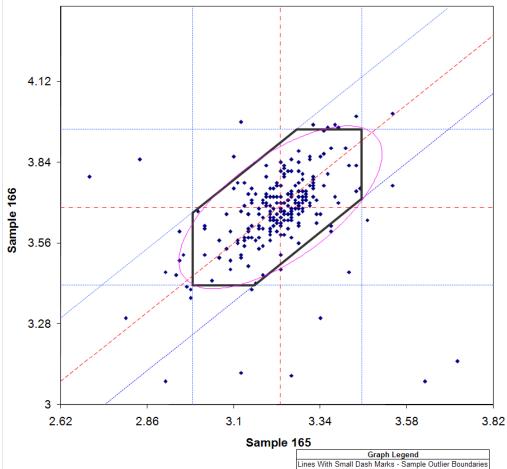
Reproducability (Sample 161)		
1s	d2s	CV%
0.06	0.18	2.33

Repro	ducability (	Sample 162)
1s	d2s	CV%
0.08	0.22	2.54



#### Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement CCRL Samples 165 and 166

Test Property: % Sulfur Trioxide (SO3), Type I/II Cement w/ Limestone



Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data:

AASHTO Materials Reference Laboratory Proficiency Sample Program CCRL Samples 165 and 166

Final Report Issued September 2007

213

Participation:

247 Total Laboratories

Laboratories Determined to be Invalid
 Laboratories Determined to be Outlier

Laboratories Determined to be Outliers Total Laboratorires Included in Analysis

 Average Results

 Sample 165 Sample 166 Average
 Average

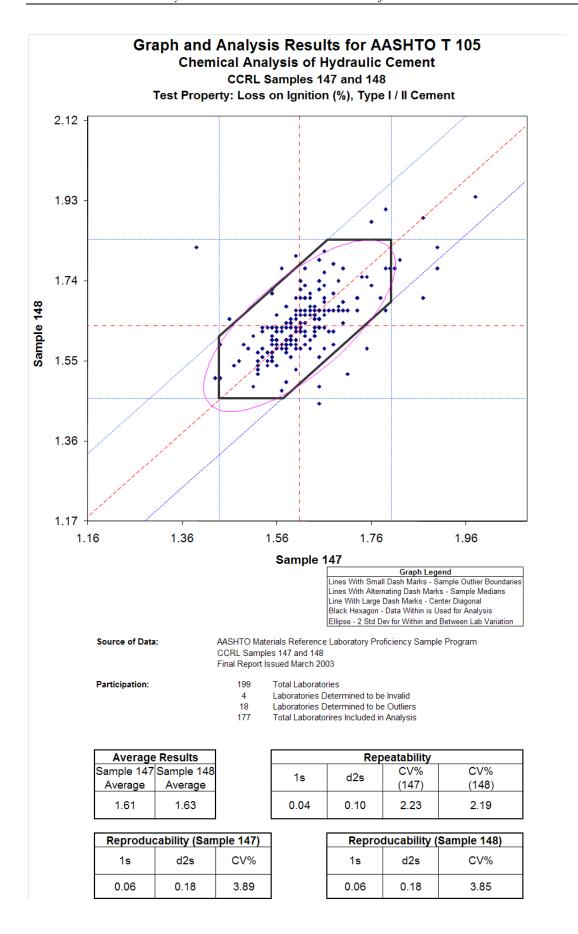
 3.23
 3.68

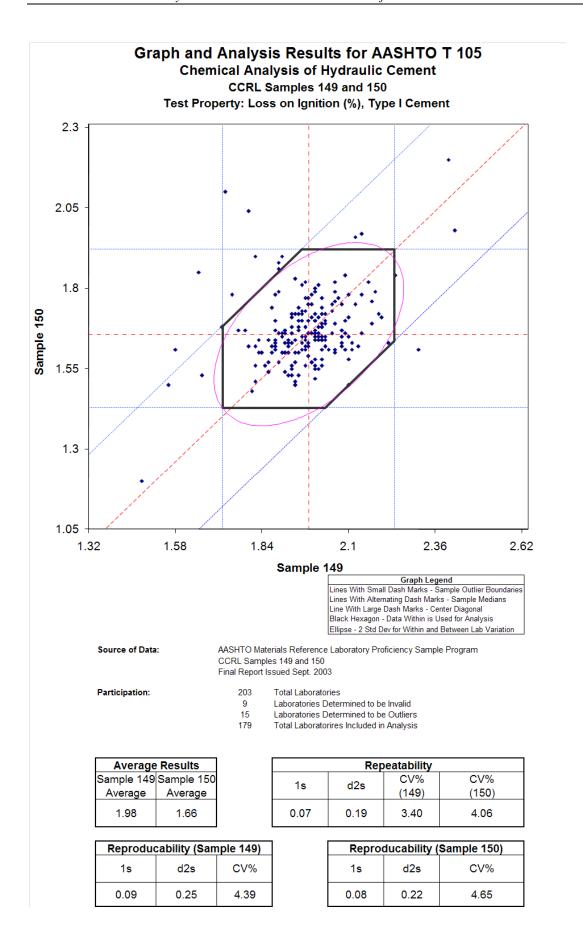
Repeatability			
1s	d2s	CV%	CV%
15	u25	(165)	(166)
0.05	0.15	1.69	1.48

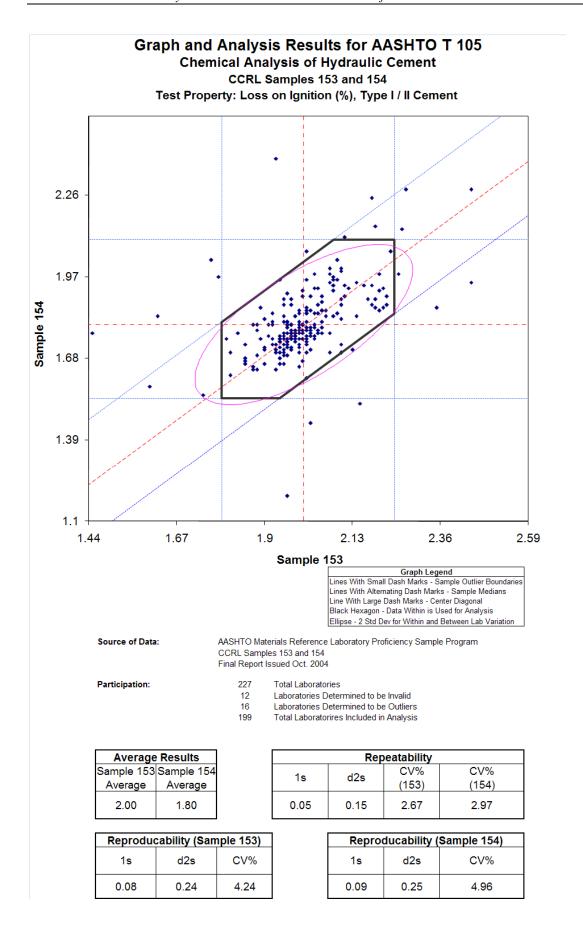
Reproducability (Sample 165)		
1s	d2s	CV%
0.08	0.23	2.48

Repro	ducability (	Sample 166)
1s	d2s	CV%
0.09	0.26	2.47

## **APPENDIX G: LOSS ON IGNITION**



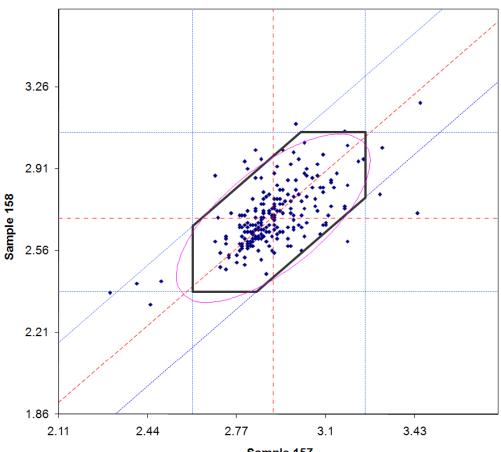




#### Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement

CCRL Samples 157 and 158

Loss on Ignition (%), Type V Cement w/ Limestone (157) & Type I / II w/ Limestone (158)



Sample 157

Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 157 and 158 Final Report Issued Oct. 2005

Participation: 229 Total Laboratories

6 Laboratories Determined to be Invalid

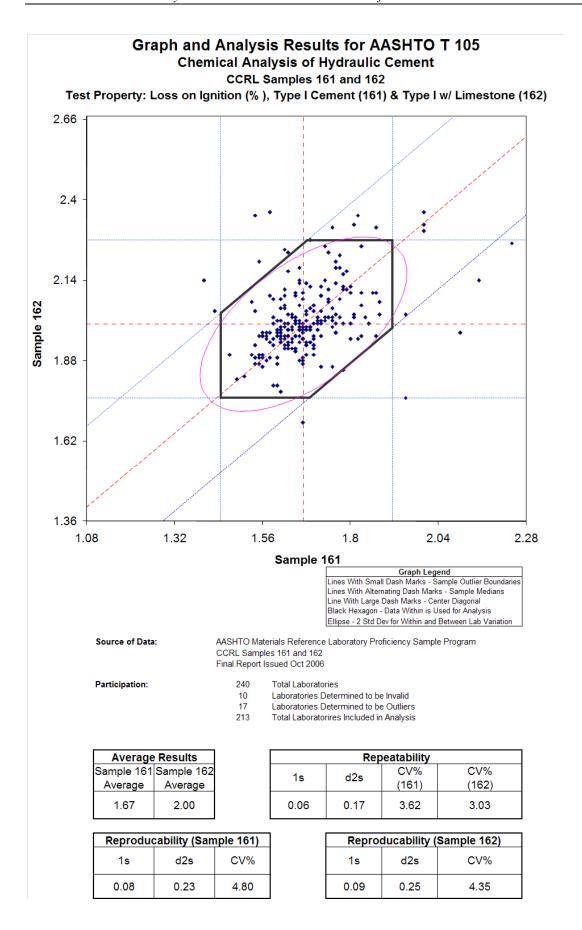
14 Laboratories Determined to be Outliers

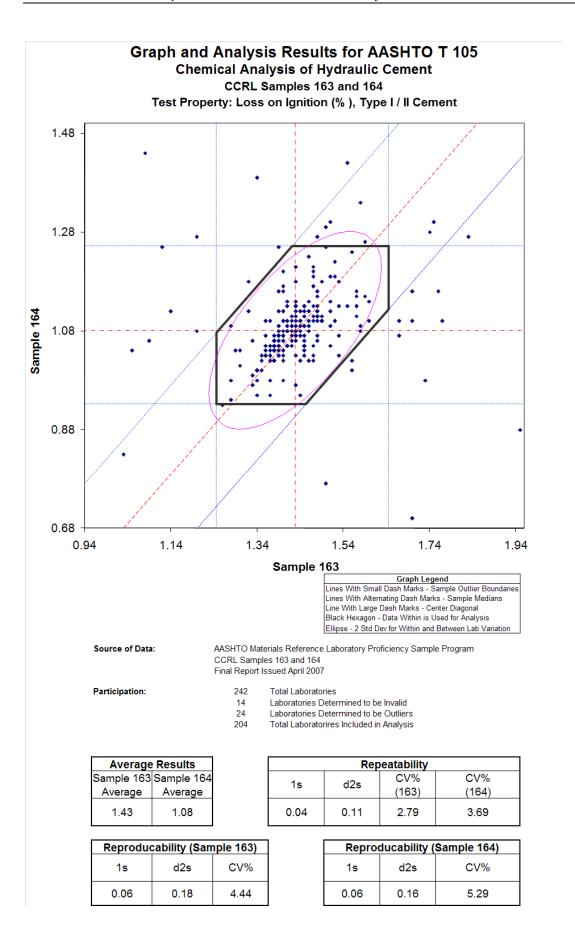
Average Results		
Sample 157 Sample 158		
Average	Average	
2.91	2.70	

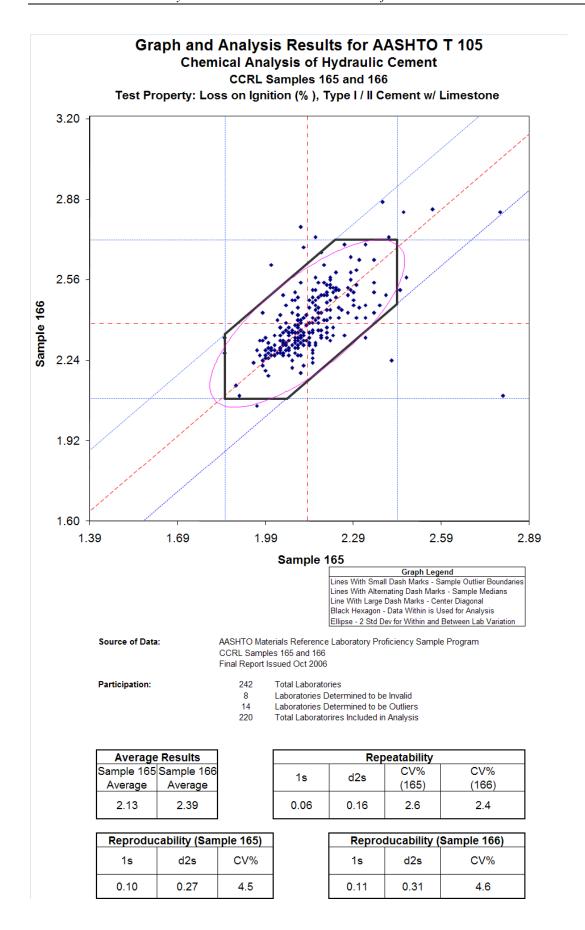
Repeatability			
1s	d2s	CV%	CV%
15	u25	(157)	(158)
0.07	0.19	2.31	2.48

Reproducability (Sample 157)		
1s	d2s	CV%
0.11	0.31	3.75

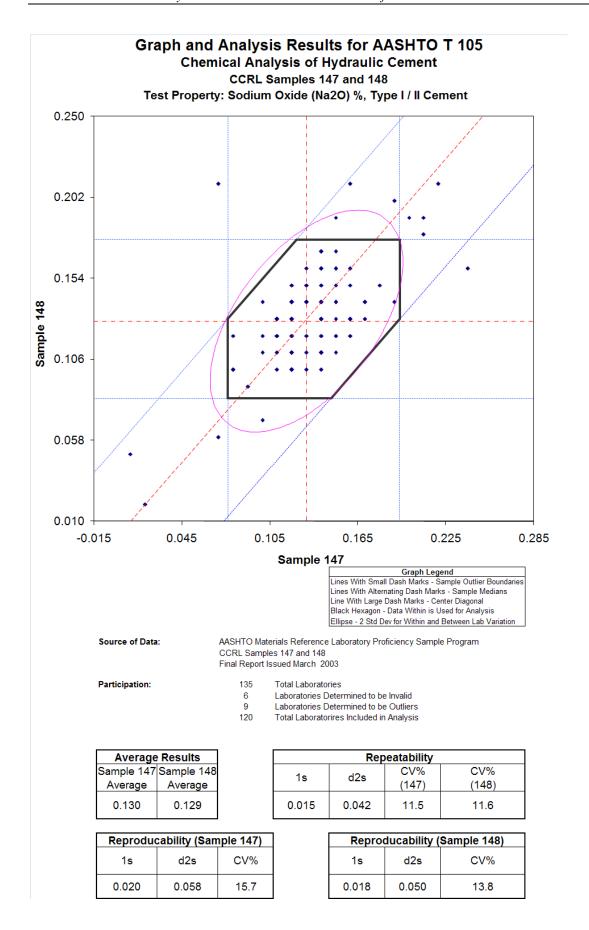
Reproducability (		Sample 158)
1s d2s		CV%
0.11	0.31	4.11

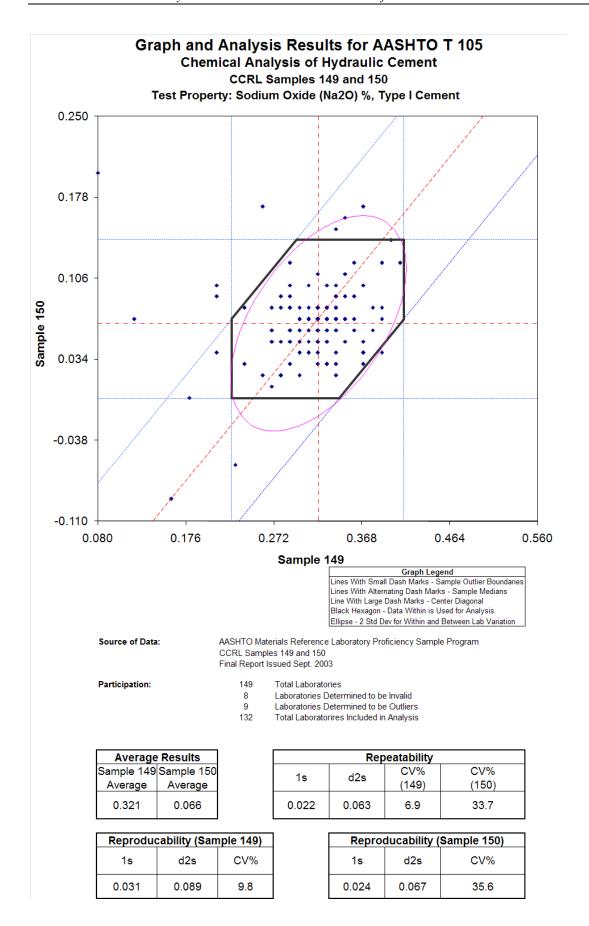


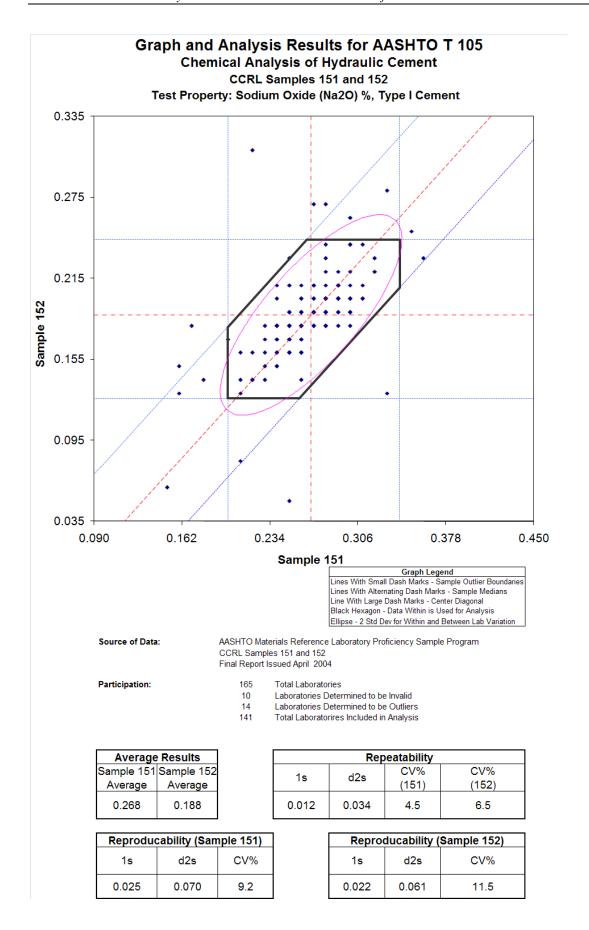


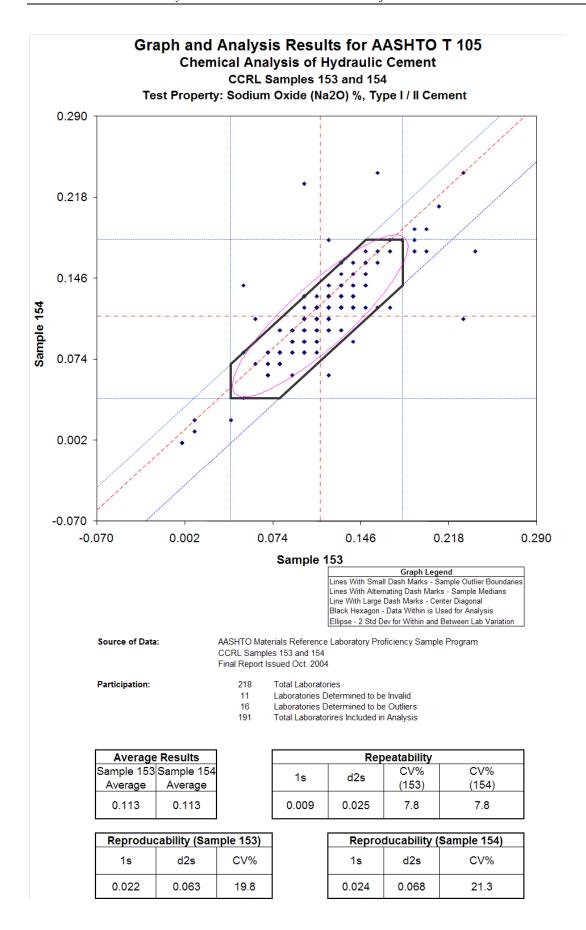


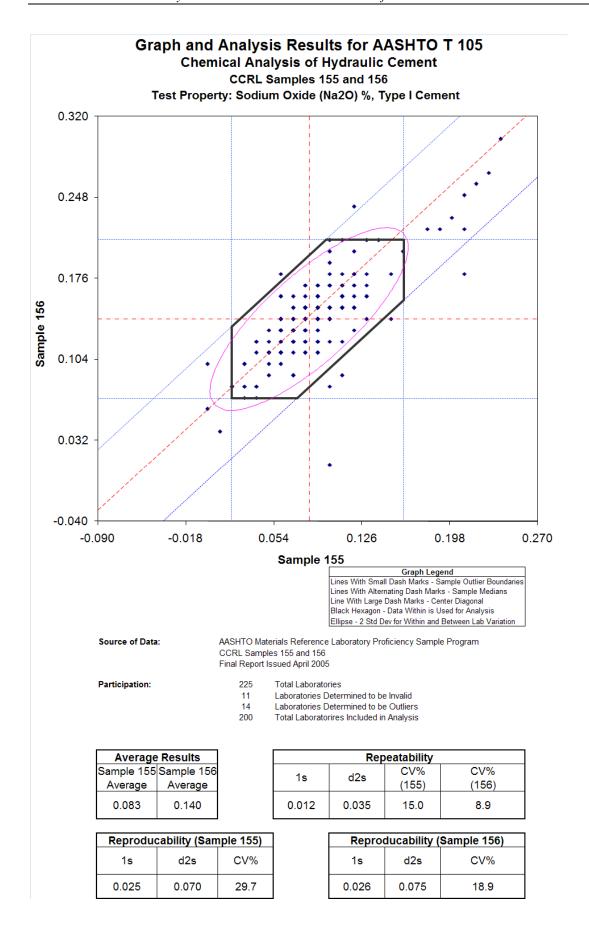
# APPENDIX H: SODIUM OXIDE (NA<sub>2</sub>O)

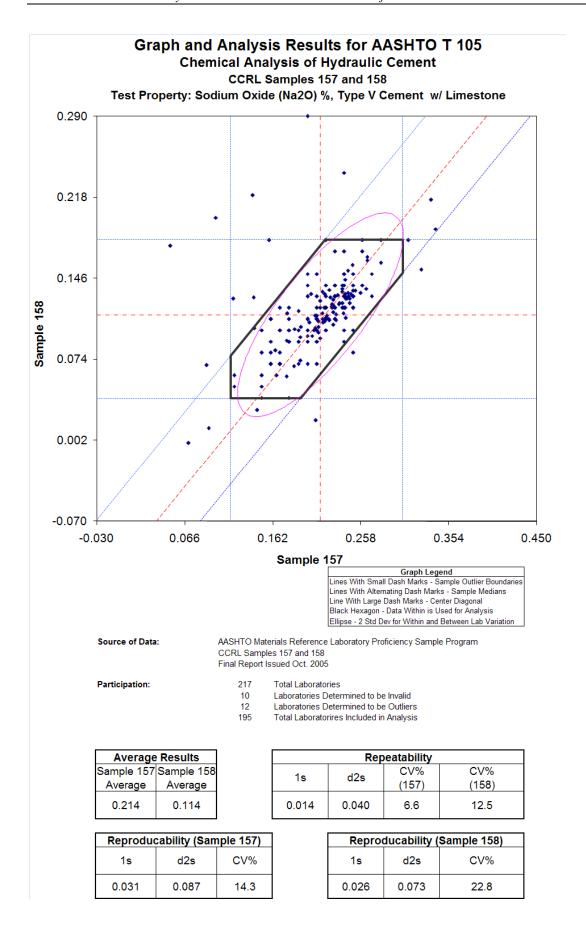


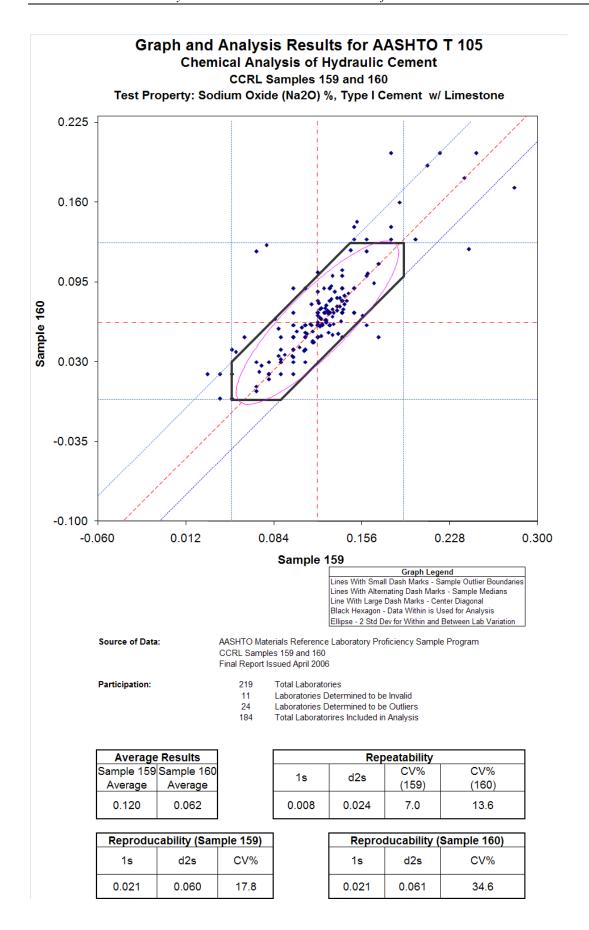


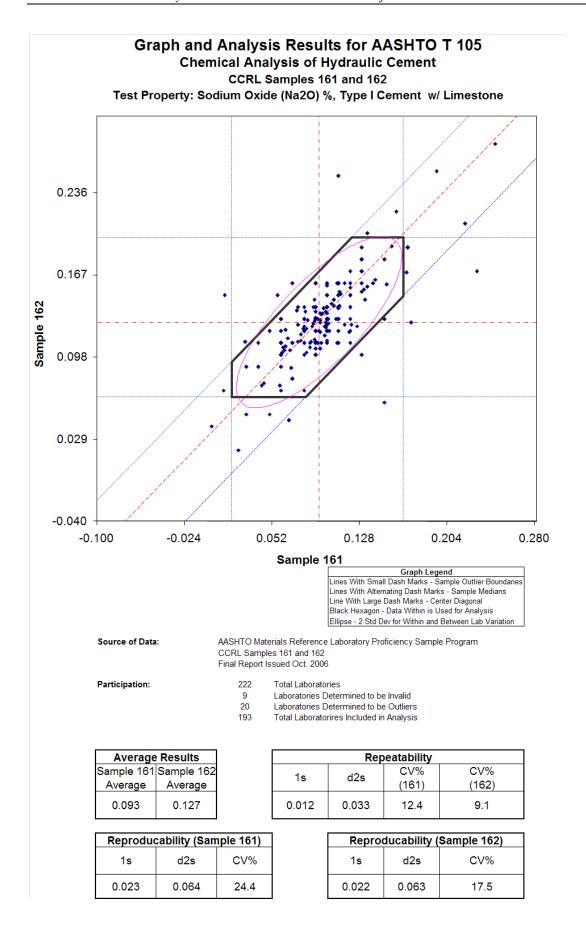


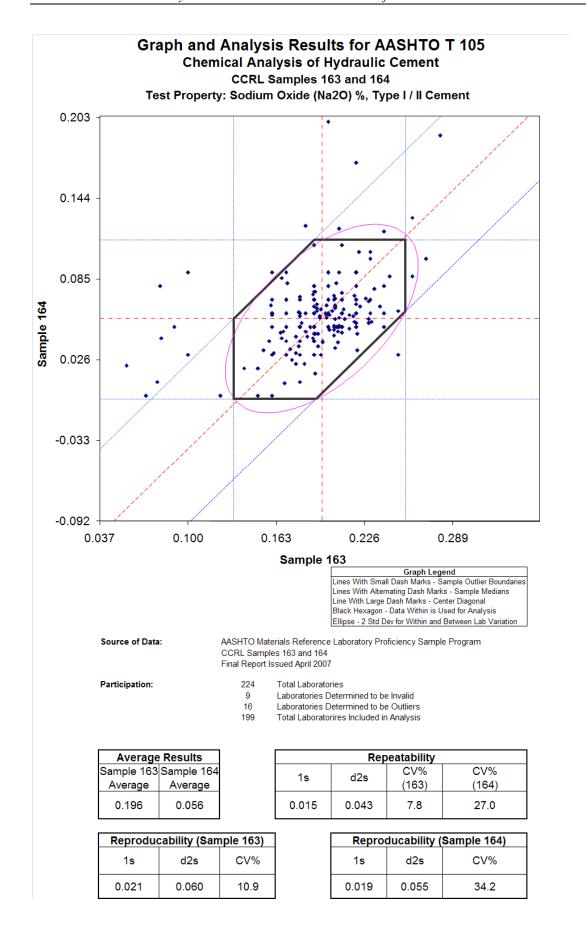


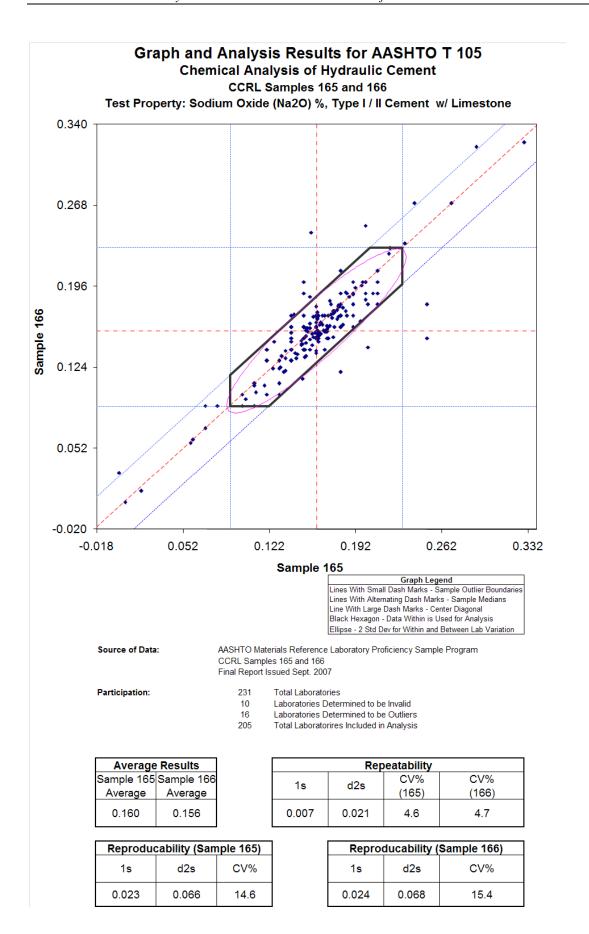




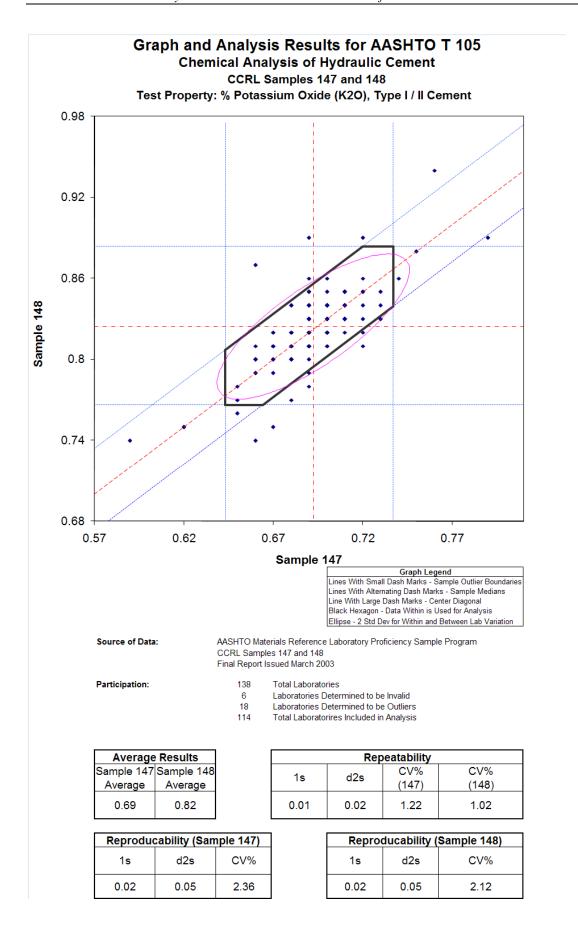


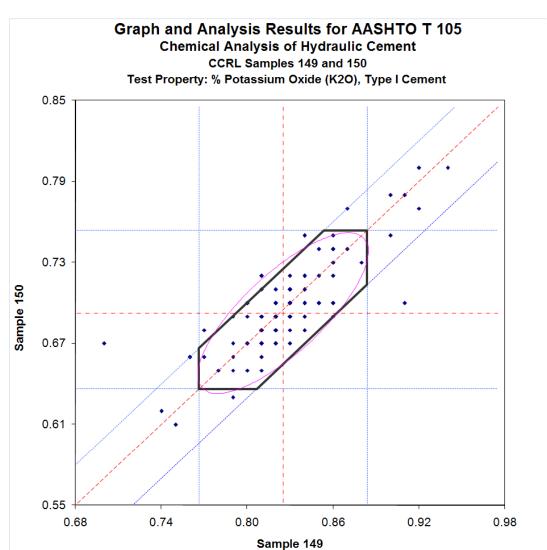






## APPENDIX I: POTASSIUM OXIDE (K<sub>2</sub>O)





imple 149

Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 149 and 150 Final Report Issued Sept. 2003

Participation: 154 Total Laboratories

9 Laboratories Determined to be Invalid

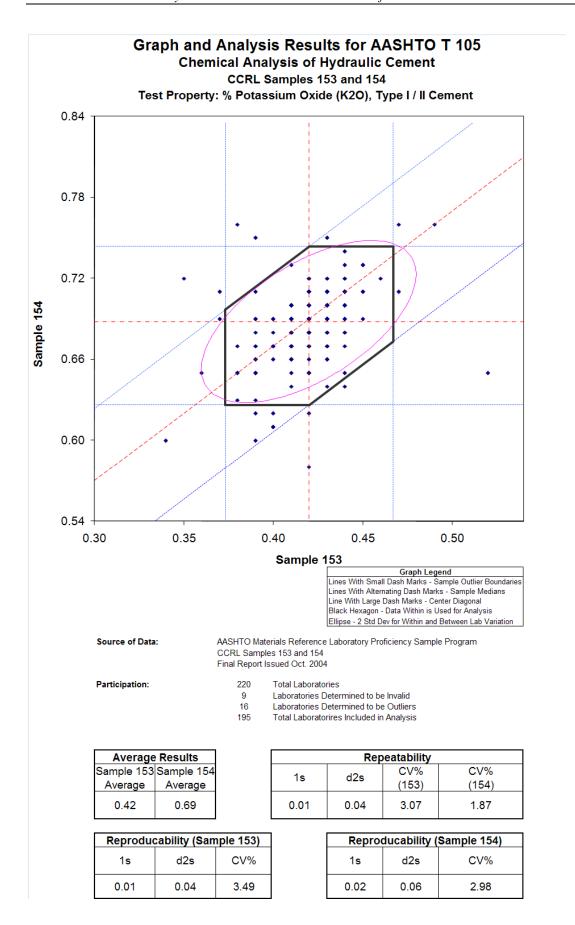
17 Laboratories Determined to be Outliers

Average Results		
Sample 149 Sample		
Average	Average	
0.83	0.69	

Repeatability			
1s	d2s	CV%	CV%
15	u25	(149)	(150)
0.01	0.03	1.15	1.37

Reproducability (Sample 149)		
1s	d2s	CV%
0.02	0.05	2.25

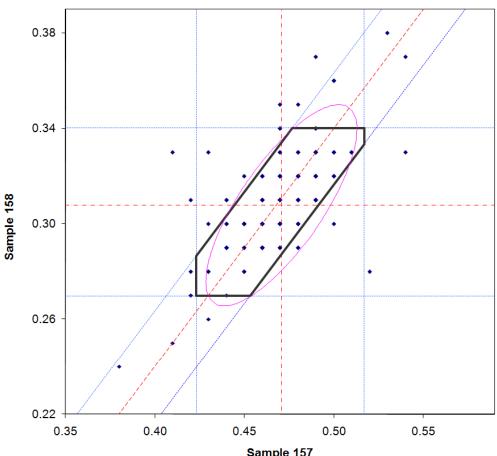
	Reproducability (		Sample 150)
			CV%
	0.02	0.05	2.72



### Graph and Analysis Results for AASHTO T 105 **Chemical Analysis of Hydraulic Cement**

CCRL Samples 157 and 158

% Potassium Oxide (K2O), Type V w/ Limestone (157) & Type I / II w/ Limestone (158)



Sample 157

Graph Legend Lines With Small Dash Marks - Sample Outlier Boundaries Lines With Alternating Dash Marks - Sample Medians Line With Large Dash Marks - Center Diagonal Black Hexagon - Data Within is Used for Analysis Ellipse - 2 Std Dev for Within and Between Lab Variatio

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 157 and 158 Final Report Issued Oct. 2005

Participation: 222 Total Laboratories

12 Laboratories Determined to be Invalid

24 Laboratories Determined to be Outliers

Average Results		
Sample 157 Sample 158		
Average	Average	
0.47	0.31	

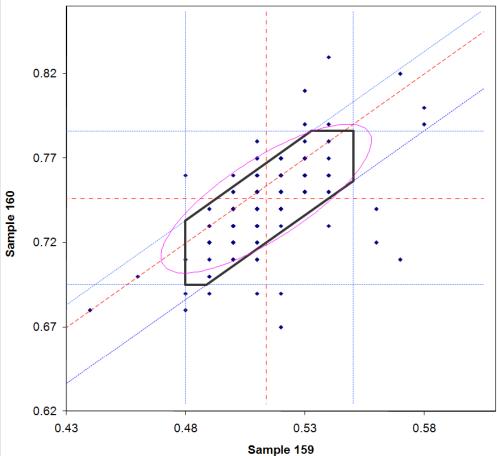
Repeatability			
1s	d2s	CV%	CV%
15	u2s	(157)	(158)
0.01	0.02	1.46	2.24

Reproducability (Sample 157)		
1s	d2s	CV%
0.01	0.04	2.99

Reproducability (		Sample 158)
1s d2s		CV%
0.01	0.03	4.00

#### Graph and Analysis Results for AASHTO T 105 **Chemical Analysis of Hydraulic Cement** CCRL Samples 159 and 160

% Potassium Oxide (K2O), Type I w/ Limestone (159) & Type I / II w/ Limestone (160)



Graph Legend Lines With Small Dash Marks - Sample Outlier Boundaries Lines With Alternating Dash Marks - Sample Medians Line With Large Dash Marks - Center Diagonal Black Hexagon - Data Within is Used for Analysis Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data:

AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 159 and 160 Final Report Issued April 2006

Participation:

224 Total Laboratories

22 Laboratories Determined to be Invalid 14 Laboratories Determined to be Outliers

Average Results		
Sample 159	Sample 160	
Average	Average	
0.51	0.75	

Repeatability			
1s d2s	CV%	CV%	
	(159)	(160)	
0.01	0.02	1.34	0.92

Reproducability (Sample 159)		
1s	d2s	CV%
0.01	0.04	2.41

Repro	ducability (	Sample 160)
1s	d2s	CV%
0.02	0.04	2.04

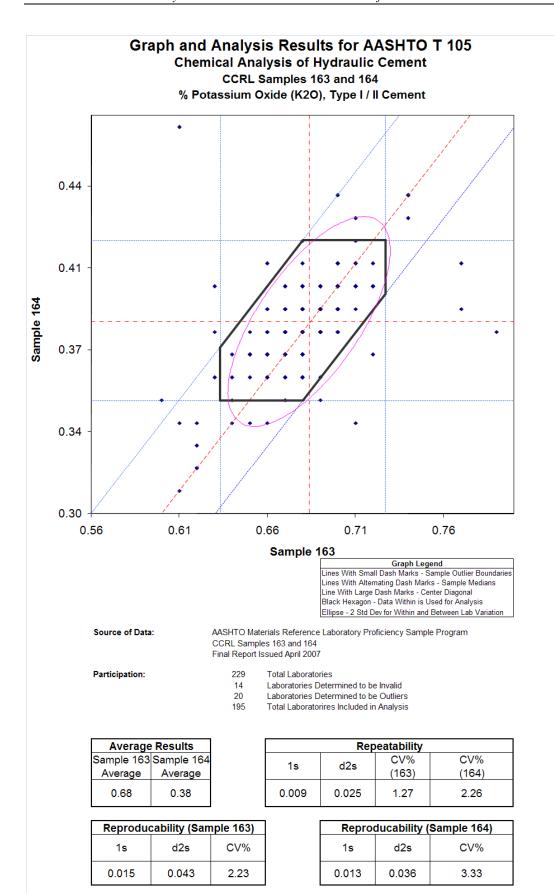
## Graph and Analysis Results for AASHTO T 105 **Chemical Analysis of Hydraulic Cement** CCRL Samples 161 and 162 % Potassium Oxide (K2O), Type I Cement (161) & Type I Cement w/ Limestone (162) 0.62 0.58 0.55 Sample 162 0.51 0.48 0.44 0.57 0.6 0.63 0.66 0.69 Sample 161 Graph Legend Lines With Small Dash Marks - Sample Outlier Boundaries Lines With Alternating Dash Marks - Sample Medians Line With Large Dash Marks - Center Diagonal Black Hexagon - Data Within is Used for Analysis Ellipse - 2 Std Dev for Within and Between Lab Variation Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program CCRL Samples 161 and 162 Final Report Issued Oct. 2006 Participation: 228 Total Laboratories 24 Laboratories Determined to be Invalid 24 Laboratories Determined to be Outliers 180 Total Laboratorires Included in Analysis

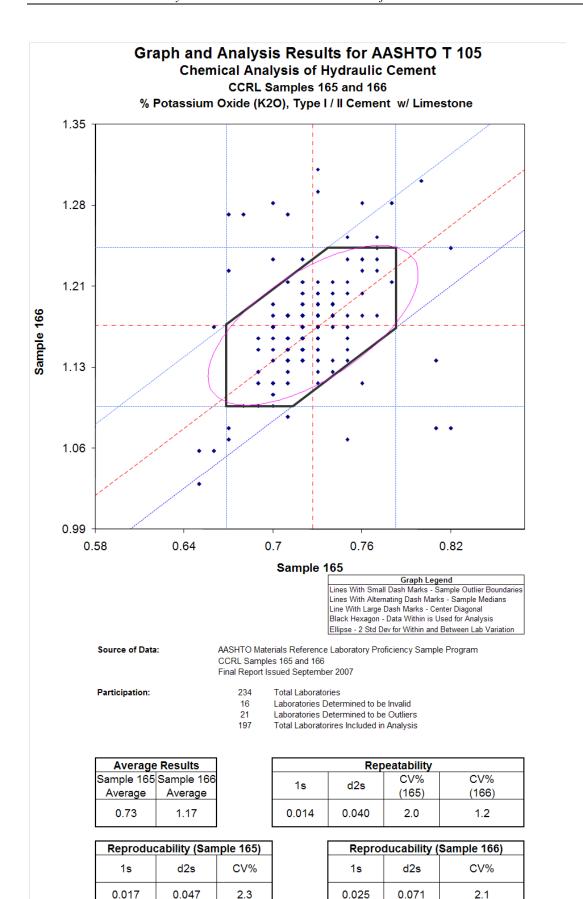
Average Results		
Sample 161	Sample 162	
Average	Average	
0.63	0.53	

Repeatability			
1s	d2s	CV% (161)	CV% (162)
0.007	0.019	1.06	1.26

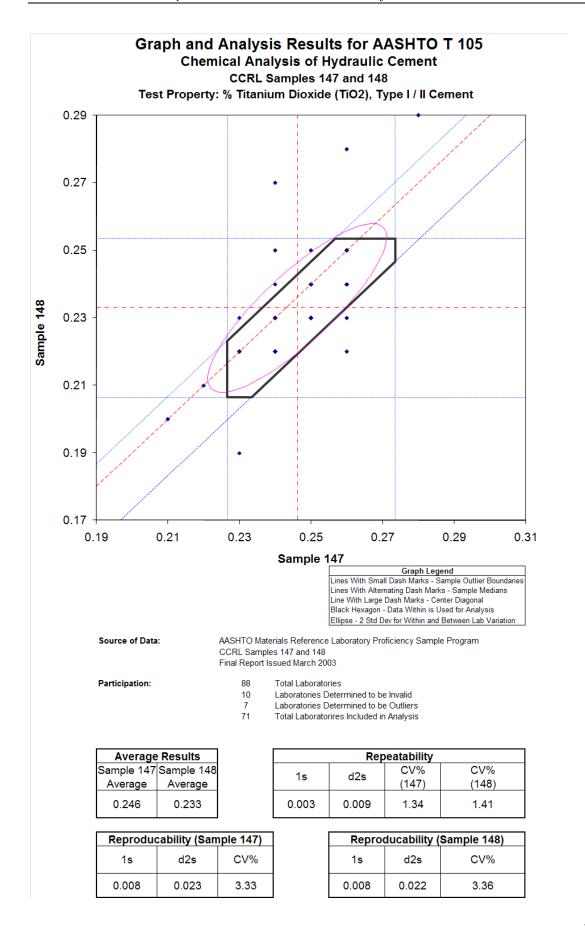
Reproducability (Sample 161)		
1s	d2s	CV%
0.011	0.030	1.67

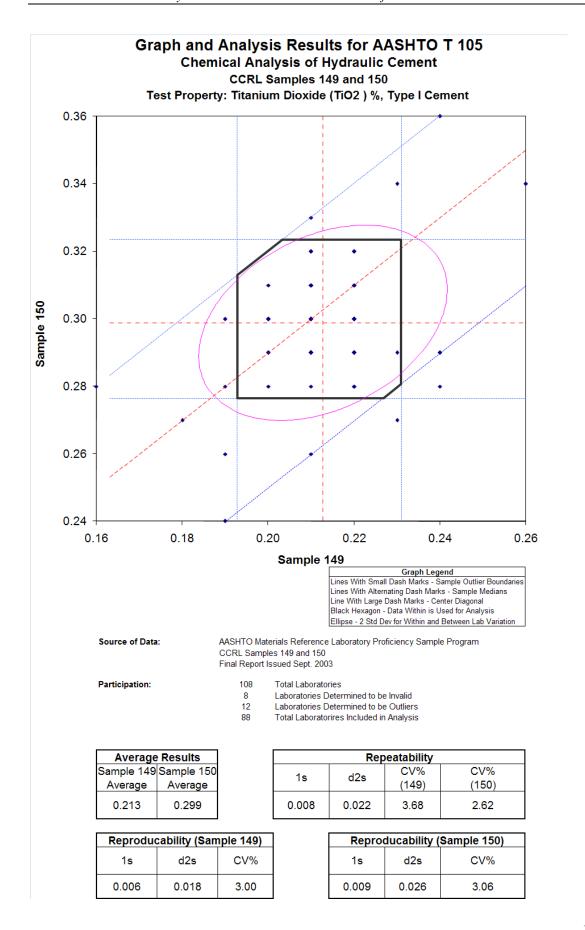
Repro	ducability (	Sample 162)
1s	d2s	CV%
0.011	0.031	2.08

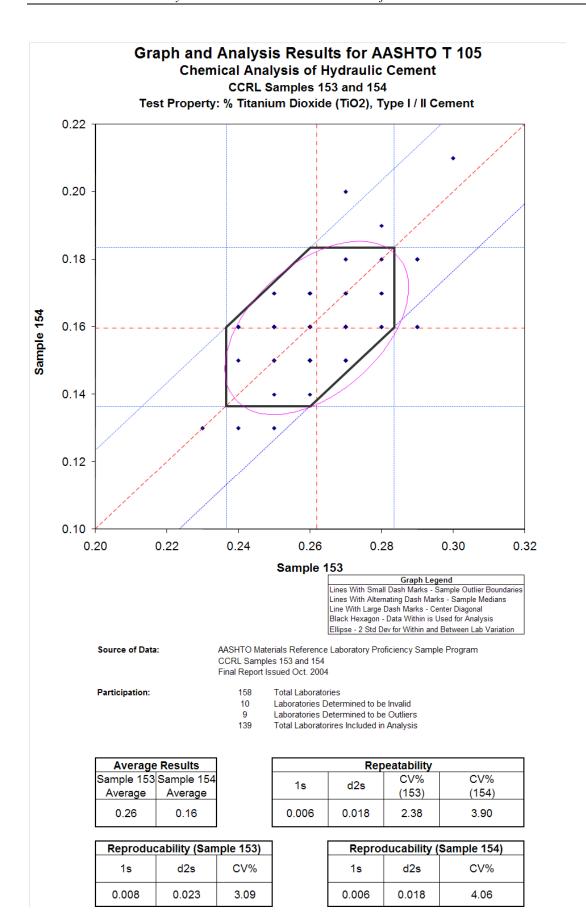


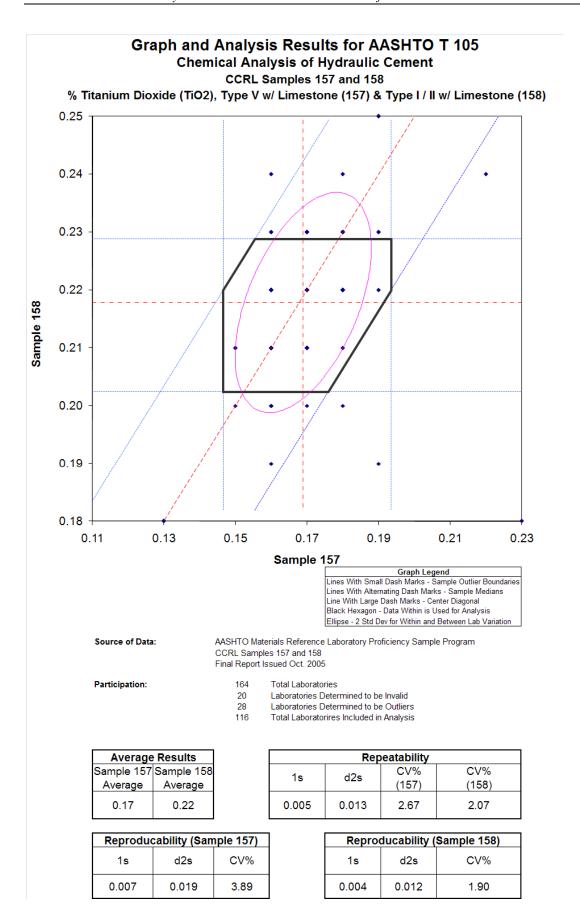


# APPENDIX J: TITANIUM DIOXIDE (TIO<sub>2</sub>)



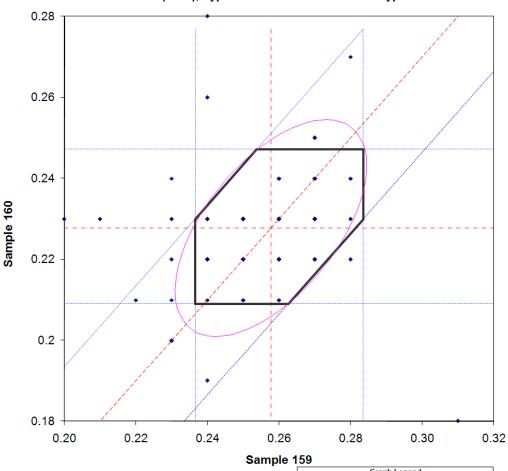






# Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement CCRL Samples 159 and 160

% Titanium Dioxide (TiO2), Type I Cement w/ Limestone & Type I / II w/ Limestone



Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 159 and 160 Final Report Issued April 2006

Participation: 173 Total Laboratories

17 Laboratories Determined to be Invalid

13 Laboratories Determined to be Outliers

Average Results			
Sample 159 Sample 160			
Average	Average		
0.26	0.23		

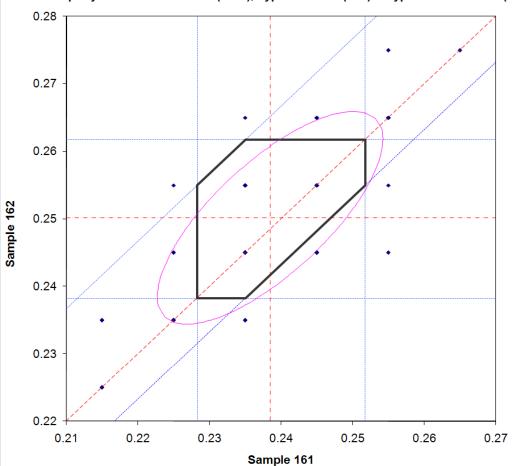
Repeatability			
1s	d2s	CV%	CV%
		(159)	(160)
0.006	0.016	2.24	2.54

Reproducability (Sample 159)			
1s	d2s	CV%	
0.009	0.025	3.41	

Reproducability (		Sample 160)
1s	d2s	CV%
0.007	0.020	3.06

#### Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement CCRL Samples 161 and 162

Test Property: % Titanium Dioxide (TiO2), Type I Cement (161) & Type I w/ Limestone (162)



Graph Legend
Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 161 and 162 Final Report Issued Oct 2006

Participation: 175 Total Laboratories

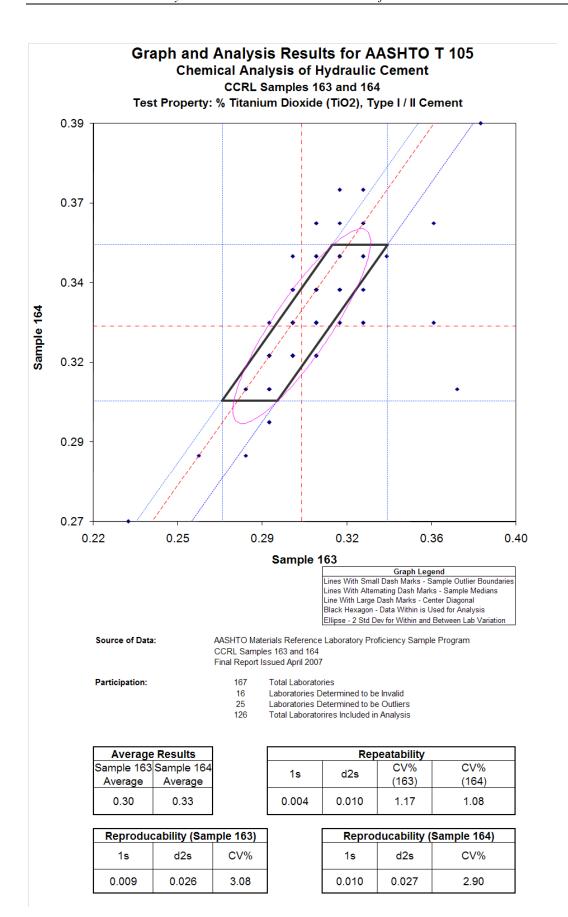
Laboratories Determined to be Invalid
 Laboratories Determined to be Outliers
 Total Laboratorires Included in Analysis

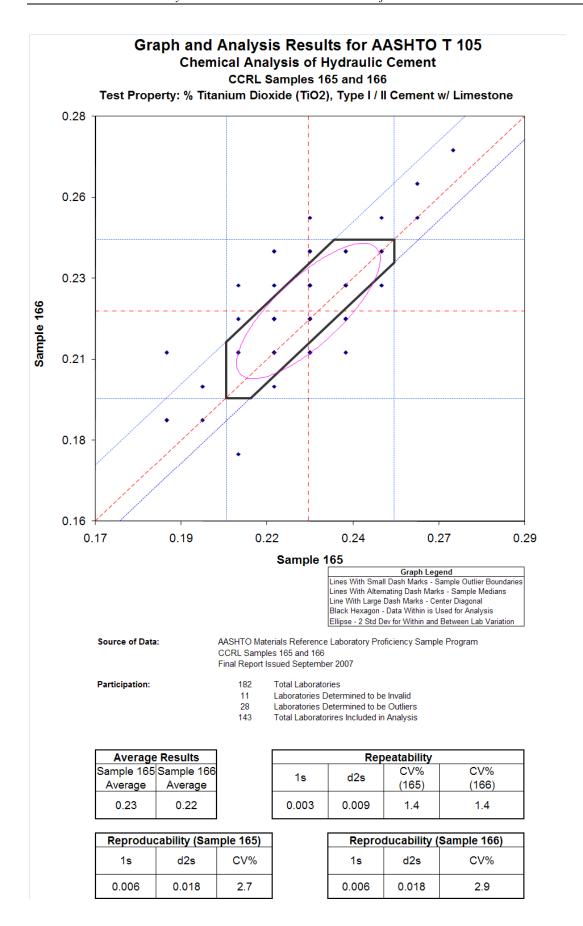
Average Results		
Sample 161	Sample 162	
Average	Average	
0.23	0.25	

Repeatability			
1s	d2s	CV%	CV%
15	uzs	(161)	(162)
0.003	0.008	1.15	1.09

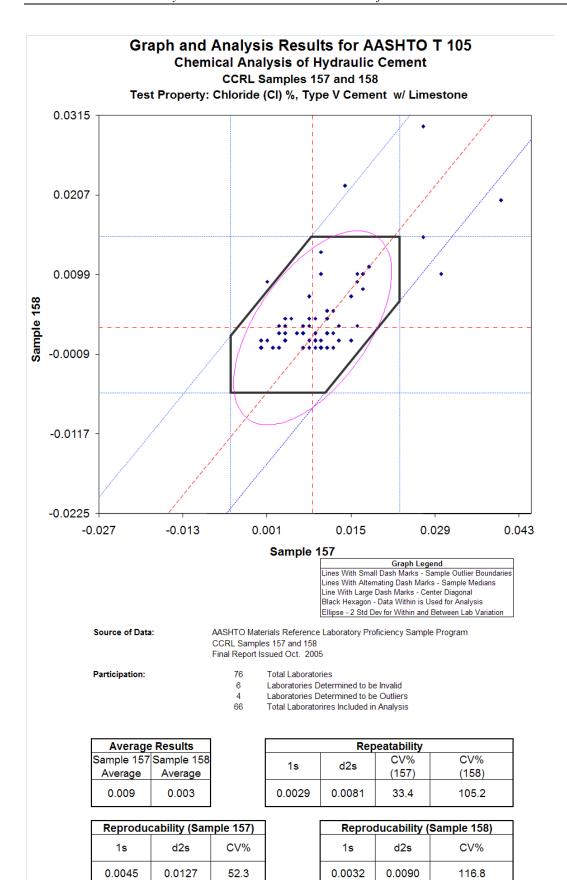
Reproducability (Sample 161)		
1s d2s CV%		CV%
0.005	0.014	2.04

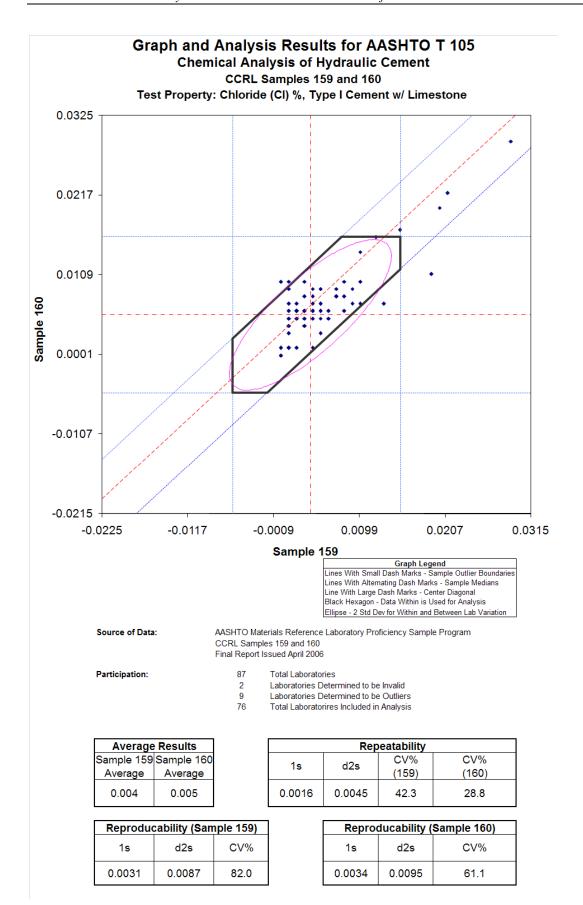
Reproducability (		Sample 162)
1s	d2s	CV%
0.005	0.014	2.05

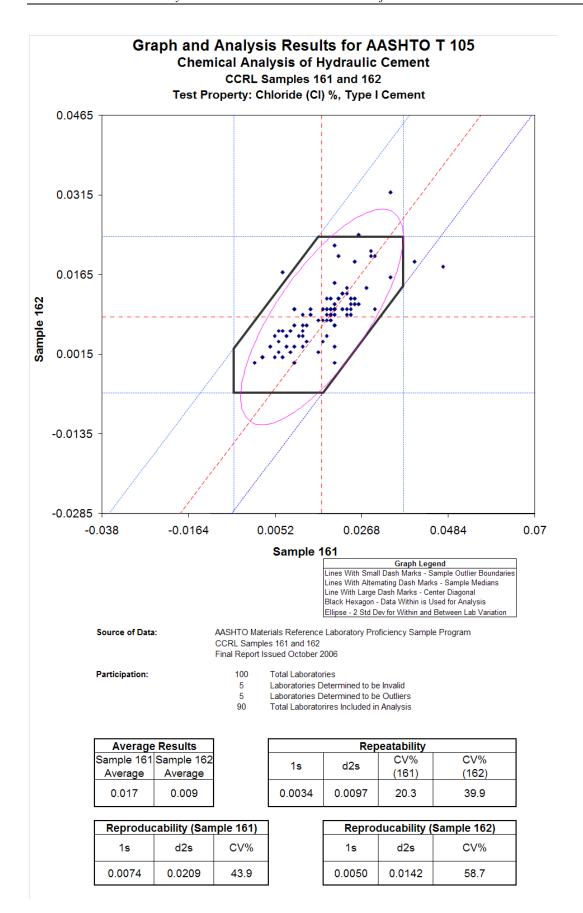


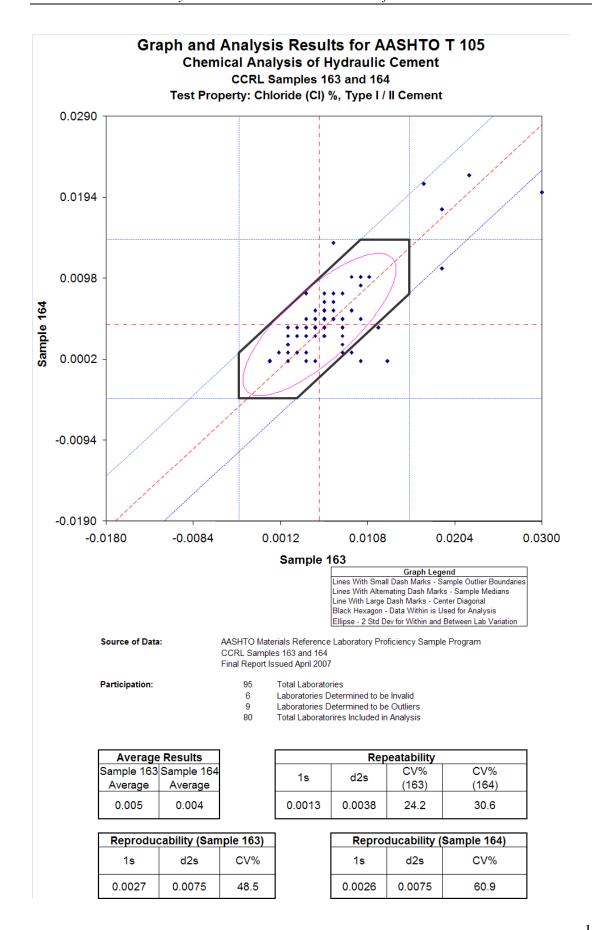


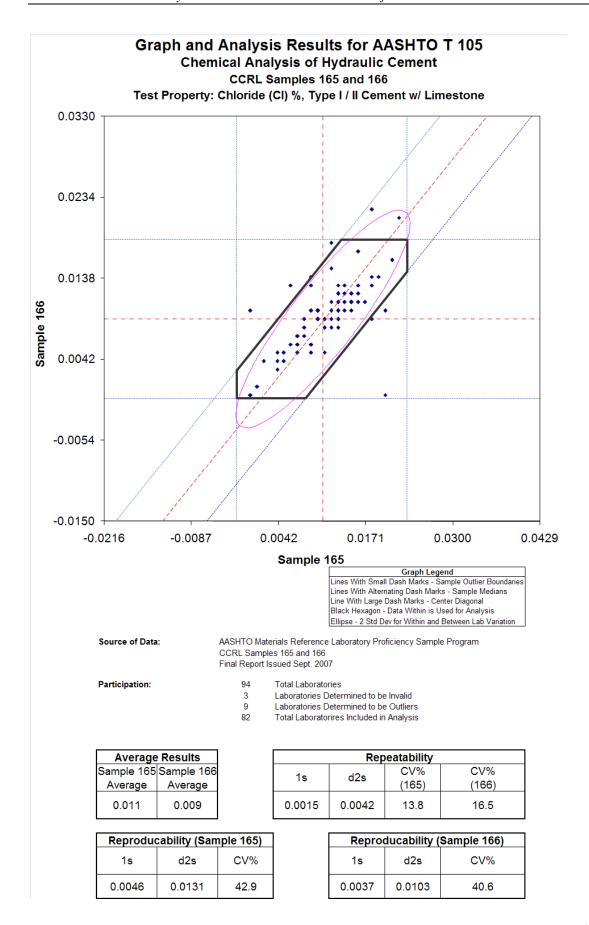
## APPENDIX K: CHLORIDE (CL)



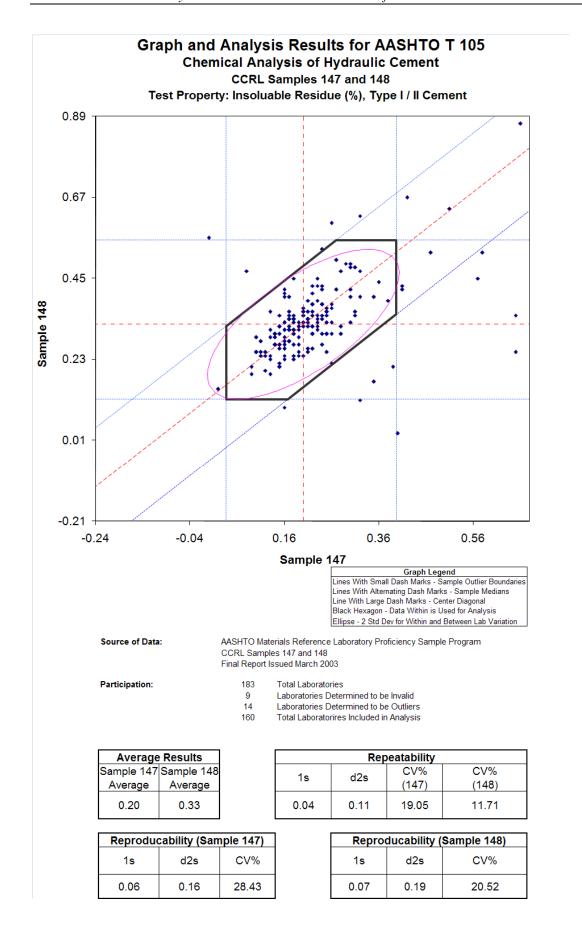


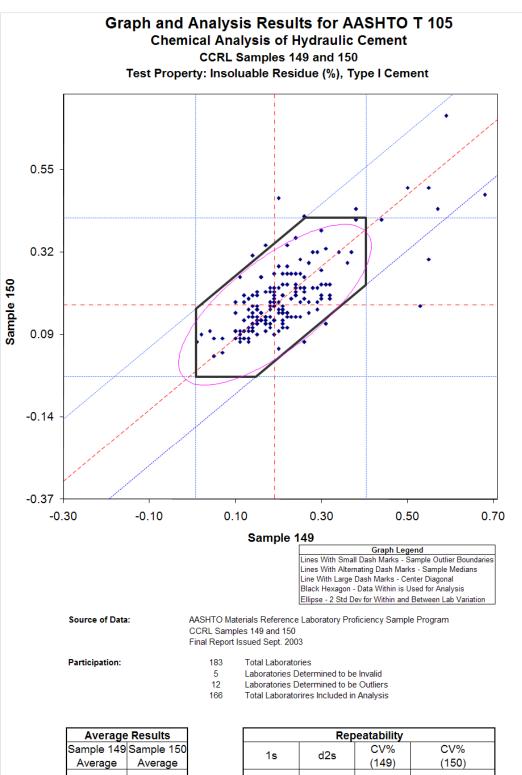






#### APPENDIX L: INSOLUABLE RESIDUE



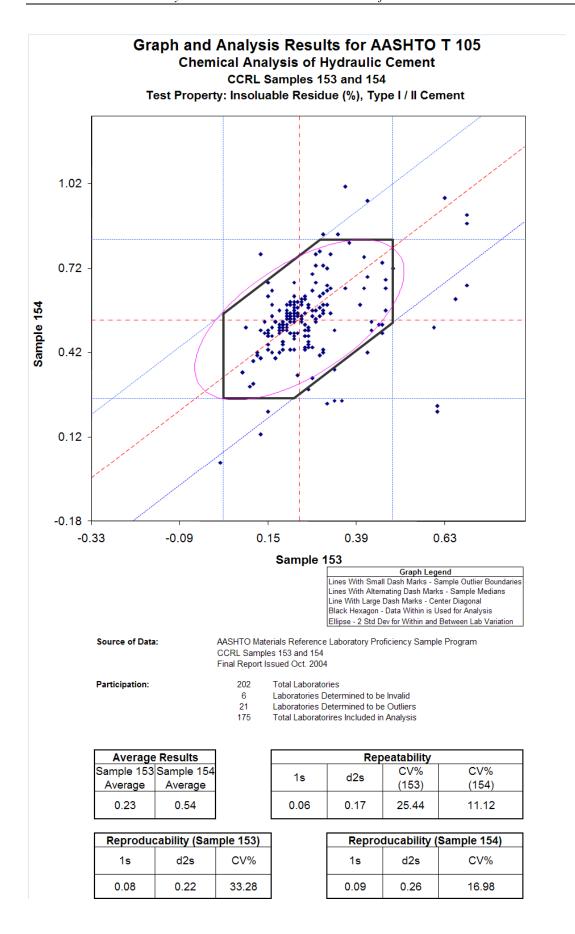


Average Results		
Sample 149 Sample 150		
Average	Average	
0.19	0.17	

Repeatability			
1s	10 420		CV%
15	d2s	(149)	(150)
0.04	0.11	20.27	22.37

Reproducability (Sample 149)		
1s	d2s	CV%
0.07	0.19	35.51

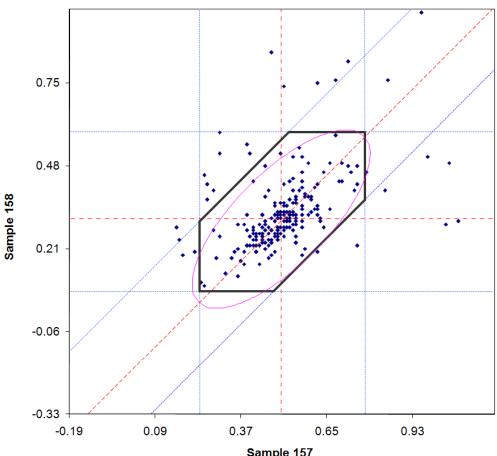
Reproducability (Sample 15		
1s	d2s	CV%
0.07	0.20	40.86



#### Graph and Analysis Results for AASHTO T 105 **Chemical Analysis of Hydraulic Cement**

CCRL Samples 157 and 158

Insoluable Residue (%), Type V Cement w/ Limestone (157) & Type I / II w/ Limestone (158)



Sample 157

Graph Legend Lines With Small Dash Marks - Sample Outlier Boundaries Lines With Alternating Dash Marks - Sample Medians Line With Large Dash Marks - Center Diagonal Black Hexagon - Data Within is Used for Analysis Ellipse - 2 Std Dev for Within and Between Lab Variatio

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 157 and 158 Final Report Issued Oct. 2005

Participation: 208 Total Laboratories

Laboratories Determined to be Invalid

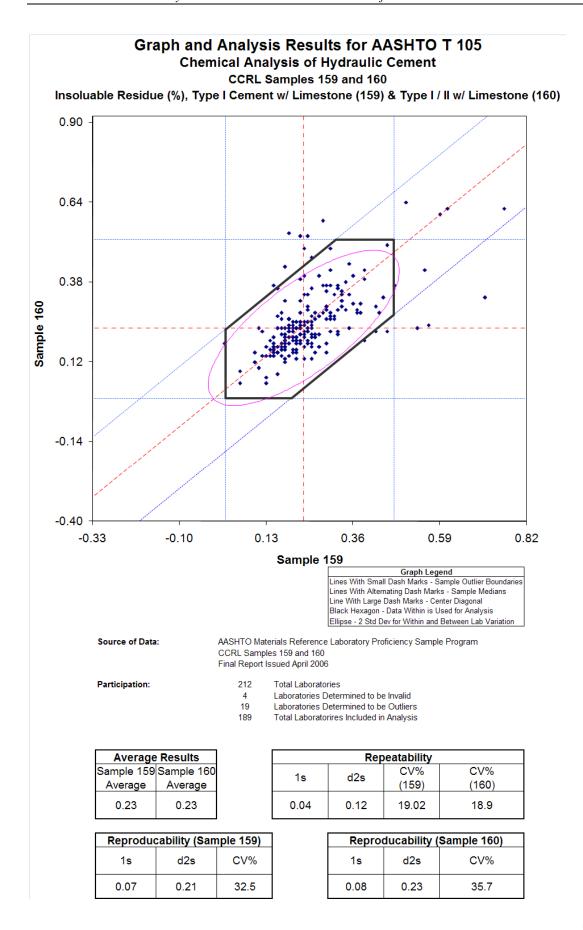
21 Laboratories Determined to be Outliers

Average Results		
Sample 157 Sample 158		
Average	Average	
0.50	0.31	

Repeatability			
1s	d2s	CV%	CV%
15	u25	(157)	(158)
0.05	0.14	10.0	16.4

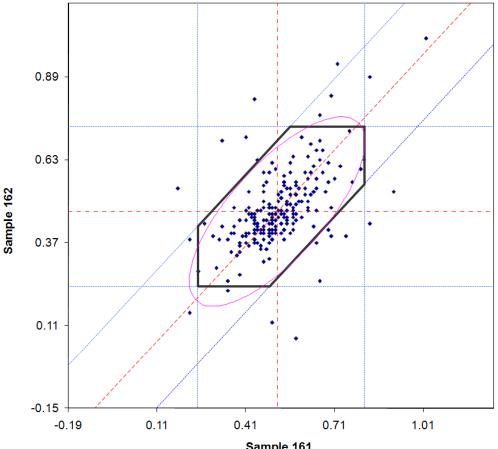
Reproducability (Sample 157)		
1s	d2s	CV%
0.09	0.26	18.5

Reproducability (Sample 158)			
1s d2s		CV%	
0.09	0.25	28.5	



#### Graph and Analysis Results for AASHTO T 105 **Chemical Analysis of Hydraulic Cement** CCRL Samples 161 and 162

Insoluable Residue (%), Type I Cement (161) & Type I Cement w/ Limestone (162)



Sample 161

Graph Legend Lines With Small Dash Marks - Sample Outlier Boundaries Lines With Alternating Dash Marks - Sample Medians Line With Large Dash Marks - Center Diagonal Black Hexagon - Data Within is Used for Analysis Ellipse - 2 Std Dev for Within and Between Lab Variatio

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 161 and 162 Final Report Issued Oct 2006

Participation: 220 Total Laboratories

Laboratories Determined to be Invalid

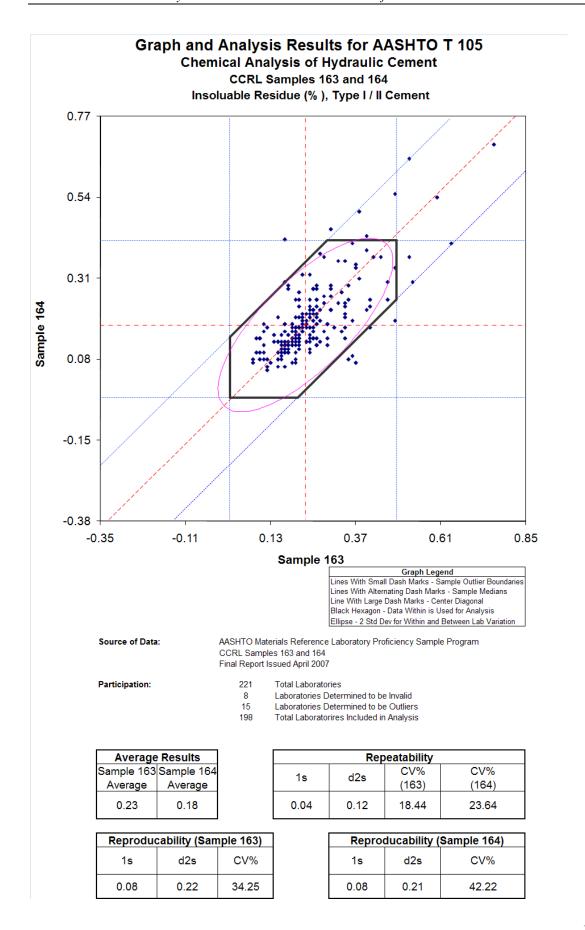
17 Laboratories Determined to be Outliers

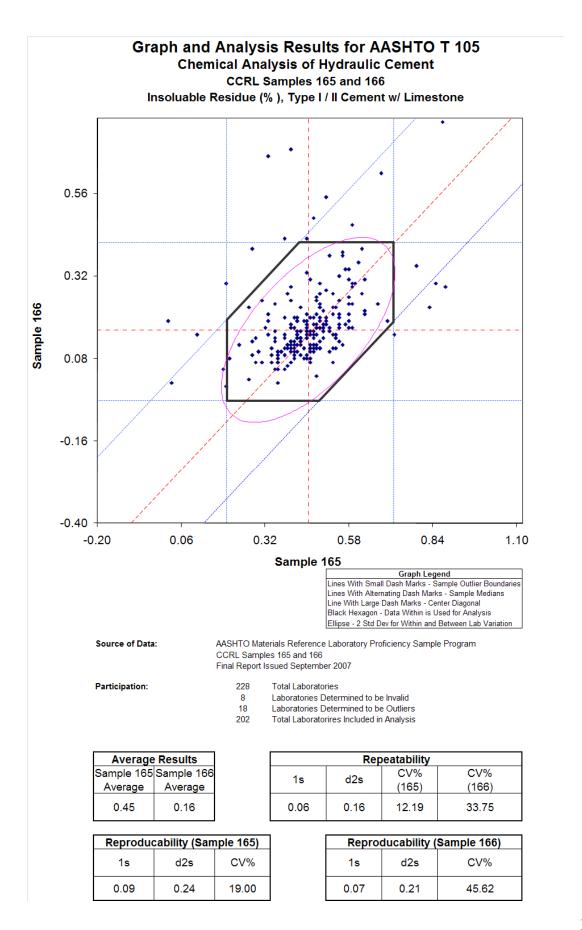
Average Results		
Sample 161	Sample 162	
Average	Average	
0.52	0.47	

Repeatability			
1s	d2s	CV%	CV%
15	u25	(161)	(162)
0.05	0.14	9.86	10.88

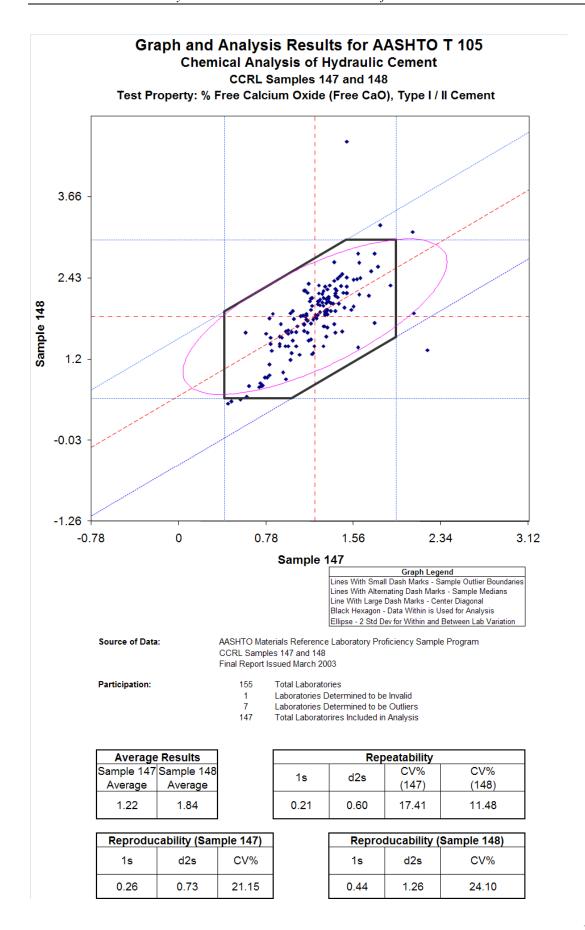
Reproducability (Sample 161)		
1s	d2s	CV%
0.10	0.27	18.67

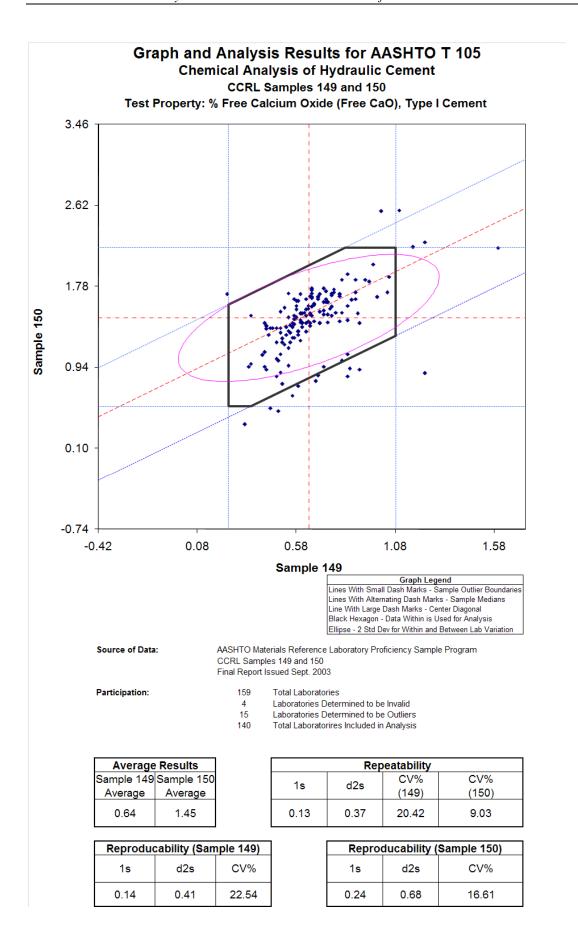
Reproducability (Sample 162)		
1s d2s		CV%
0.09	0.25	18.81

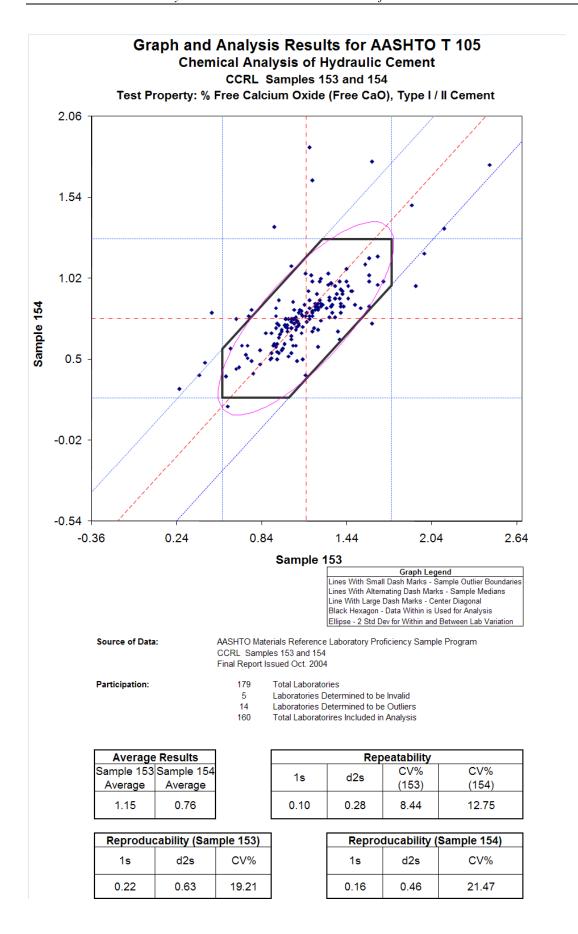




## APPENDIX M: FREE CALCIUM OXIDE (CX)

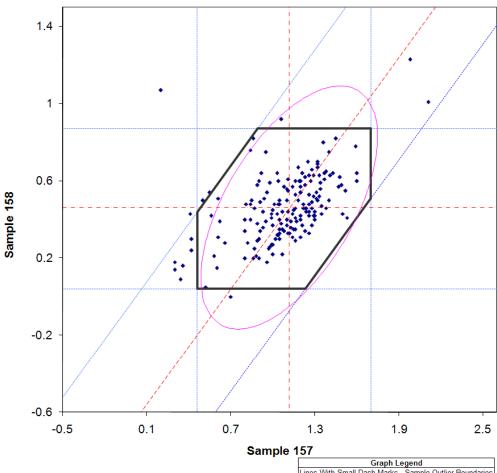






## Graph and Analysis Results for AASHTO T 105 Chemical Analysis of Hydraulic Cement CCRL Samples 157 and 158

% Free Calcium Oxide (Free CaO), Type V w/ limestone (157) & Type I / II w/ limestone (158)



Lines With Small Dash Marks - Sample Outlier Boundaries
Lines With Alternating Dash Marks - Sample Medians
Line With Large Dash Marks - Center Diagonal
Black Hexagon - Data Within is Used for Analysis
Ellipse - 2 Std Dev for Within and Between Lab Variation

Source of Data: AASHTO Materials Reference Laboratory Proficiency Sample Program

CCRL Samples 157 and 158 Final Report Issued Oct. 2005

Participation: 177 Total Laboratories

Laboratories Determined to be Invalid

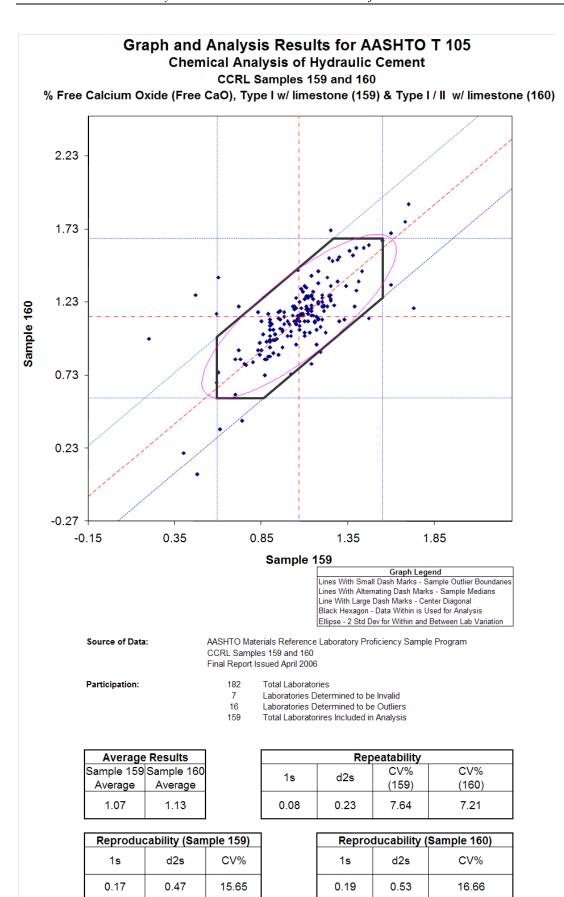
12 Laboratories Determined to be Outliers

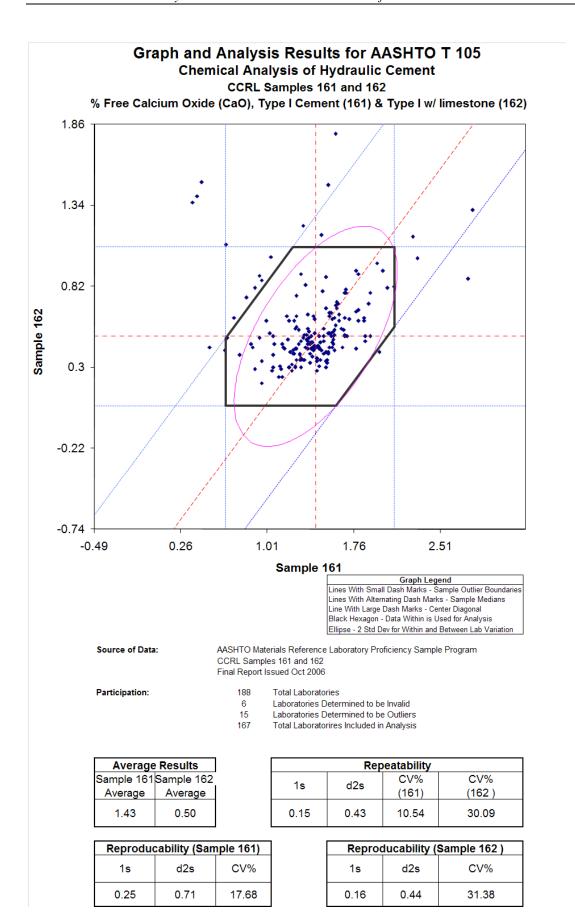
Average Results		
Sample 157 Sample 158		
Average	Average	
1.12	0.46	

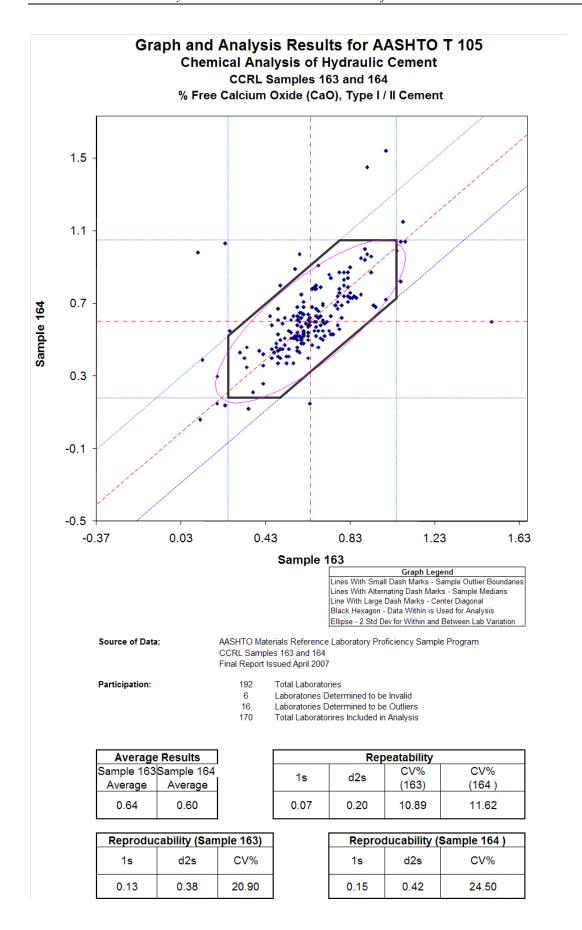
Repeatability			
1s	d2s	CV%	CV%
15	u25	(157)	(158)
0.14	0.39	12.18	29.42

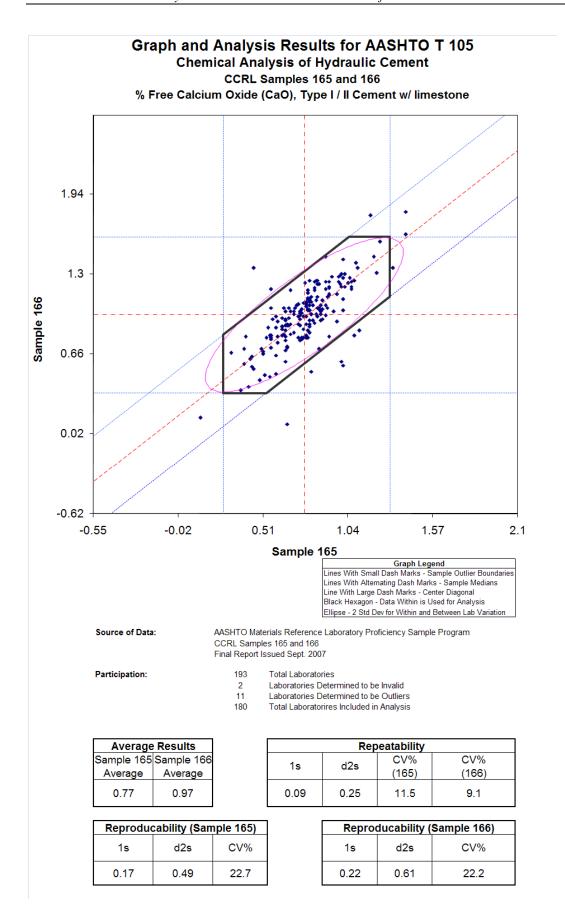
Reproducability (Sample 157)		
1s	d2s	CV%
0.22	0.62	19.66

Reproducability (Sample 158)			
1s d2s		CV%	
0.15	0.41	31.69	









## APPENDIX N: CARBON DIOXIDE (CO<sub>2</sub>)

