Recommended Precision Statements for AASHTO Standard Methods of Test T22, T 104, T 105, T 154, T 186, and T 242

DETAILS

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RECOMMENDED PRECISION STATEMENTS FOR AASHTO STANDARD METHODS OF TEST T 22, T 104, T 105, T 154, T 186, AND T 242

This digest summarizes key findings obtained between January 2007 and June 2009 under continuing NCHRP Project 9-26A, "Data Mining and Interlaboratory Studies to Prepare Precision Statements for AASHTO Standard Test Methods." NCHRP Project 9-26A was conducted by the AASHTO Materials Reference Laboratory under the direction of the Principal Investigator, Dr. Haleh Azari. This digest is based on task reports co-authored by Dr. Azari and Messrs. Ronald Holsinger, Robert Lutz, and Peter Spellerberg; these task reports are available online as *NCHRP Web-Only Documents 139, 140, 141,* and *142*.

INTRODUCTION

The objective of NCHRP Project 9-26A is to recommend new or updated precision statements of AASHTO standard methods of test designated by the technical sections of the AASHTO Highway Subcommittee on Materials (HSOM). To meet this objective, NCHRP Project 9-26A uses both data mining techniques and interlaboratory studies (as defined in ASTM D 6631, Standard Guide for Committee D01 for Conducting an Interlaboratory Practice for the Purpose of Determining the Precision of a Test Method). The project is a continuing, open-ended effort; reports are published in the form of NCHRP Web-Only Documents (WOD) as tasks related to individual standard methods are completed. Precision statements and supporting results are provided to AASHTO HSOM for review and possible adoption.

This Research Results Digest summarizes the findings of research conducted between January 2007 and June 2009 in Project 9-26A to recommend new or updated precision statements of the AASHTO standard methods of test shown in Table 1. These specific precision statements were developed through statistical analysis of multi-year results obtained from (1) the Proficiency Sample Programs (PSP) of the AASHTO Materials Reference Laboratory (AMRL) and the ASTM Cement and Concrete Reference Laboratory (CCRL) and (2) calibration testing of state friction measurement systems at field test centers at the Texas Transportation Institute, College Station, Texas, and the Transportation Research Center, East Liberty, Ohio.

A complete report of the development of each precision statement is presented in the WODs (*1, 2, 3, 4*) listed in Table 1.

FINDINGS

Data Sets

Precision statements for methods of test T 22, T 154, and T 186 were developed through an analysis of 24 data sets collected from laboratories participating in the physical analysis of hydraulic cement paste and measurement of the compressive properties of hydraulic cement concrete in the CCRL PSP between 2003 and 2007. These data

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Table 1 Methods of test and web-only documents

AASHTO Standard Method of Test	NCHRP Web-Only Document
T 22, Compressive Strength of Cylindrical Concrete Specimens	140
T 104, Soundness of Aggregate by Use of Sodium Sulfate or Magnesium Sulfate	141
T 105, Chemical Analysis of Hydraulic Cement	139
T 154, Time of Setting of Hydraulic Cement Paste by Gillmore Needles	140
T 186, Early Stiffening of Hydraulic Cement (Paste Method)	140
T 242, Frictional Properties of Paved Surfaces Using a Full-Scale Tire	142

sets reflect a wide range of test data for the cement types included in the scope of the CCRL PSP.

The precision statement for method of test T 104 was developed through an analysis of six data sets collected from laboratories participating in the analysis of sulfate soundness of coarse and fine aggregate in the AMRL PSP between the years 2003 and 2009.

The precision statement for method of test T 105 was developed through an analysis of 10 data sets collected from laboratories participating in the chemical analysis of hydraulic cement in the CCRL PSP between the years 2003 and 2007.

The precision statement for methods of test T 242 was developed through an analysis of friction data collected during the calibration of state friction measurement systems at Texas Transportation Institute and Transportation Research Center field test centers. Two sets of data were analyzed: "Initial" and "Final" as referred to by TTI or "Arrival" and "Departure" as referred to by TRC. The Initial or Arrival set was collected when the state systems arrived at the centers; the Final or Departure set was collected after adjustments were made to the state systems to put them into compliance with ASTM E 274, Skid Resistance of Paved Surfaces Using a Full-Scale Tire, which is equivalent to AASHTO T 242. The TTI Initial and Final data sets consisted, respectively, of 288 friction numbers from 12 run-repeats of 8 state friction measurement systems and 1,260 friction numbers from 12 run-repeats of 12 state friction measurement systems. The TRC Arrival and Departure data sets consisted, respectively, of 1,296 friction numbers from 12 run-repeats of 12 state friction measurement systems and 5,400 friction numbers from 12 skids of state friction measurement systems in 50 different configurations (left, right, or both wheels with either ribbed, smooth, or both tire types).

Data Analysis

The PSP data were analyzed with a technique developed by AMRL in Phase 3 of NCHRP Project 9-26 (*5*). This technique is a four-step procedure 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 multi-laboratory estimates of precision.

Analysis of the friction data was based on the method in ASTM E 691, Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method. Prior to the analysis, partial data sets were eliminated by following the procedures described in ASTM E 691 in determining repeatability (S_r) and reproducibility (S_R) estimates of precision. Data exceeding critical *h* and *k* values were eliminated as described in Section 15.6 of the test method; these same data were also eliminated from any smaller subsets analyzed.

New Precision Statements and Comparison with Existing Statements

AASHTO Standard Method of Test T 22

The 7-day compressive strength of hydraulic cement concrete was analyzed for one set of 4 in. × 8 in. and five sets of 6 in. × 12 in. proficiency sample pairs. Precision estimates are based, where appropriate, on either the coefficients of variation (CV%) or the pooled standard deviation (1s) values.

New criteria developed in this research for judging the acceptability of compressive strengths obtained by AASHTO T 22 are given in Table 2. The figures given in the second column of Table 2 are the standard deviations that were found to be appropriate for the materials and conditions of test described in the first column of the table. The figures in the third

Table 2 Precision estimates of compressive strength of cylindrical concrete specimens

Condition of Test and Test Property	Coefficient of Variation (1s), Percent of Average*	Acceptable Range of Two Test Results (d2s), Percent of Average*
Single-Operator Precision:		
6 × 12 in. (150 × 300 mm)	4.7	13.2
4 × 8 in. (100 × 200 mm)	4.3	12.1
Multi-Laboratory Precision:		
6 × 12 in. (150 × 300 mm)	8.1	23.0
4 × 8 in. (100 × 200 mm)	7.6	21.6

*These values represent the 1s and d2s limits described in ASTM Practice C670.

NOTE: The precision estimates given in Table 2 are based on the analysis of test results from one pair of 4 in. × 8 in. and five pairs of 6 in. × 12 in. CCRL hydraulic concrete proficiency samples. The data analyzed consisted of results from 267 laboratories for the 4 in. × 8 in. samples and 910 to 1,002 laboratories for each of the 6 in. × 12 in. sample pairs. The analysis included 4 in. × 8 in. samples with an average compressive strength of 3,950 psi and 6 in. × 12 in. samples with an average compressive strength of 3,851 psi to 4,812 psi.

column are the limits that should not be exceeded by the difference between the results of two properly conducted single-operator or multi-laboratory tests. The bias of AASHTO T 22 was not determined because no comparison with a material having an accepted reference value was conducted.

Table 3 compares the new and existing precision estimates for AASHTO T 22; the new repeatability and reproducibility standard deviations are larger than the existing precisions. It is not clear if this difference is due to a change in the CCRL reference material or changes in the method of test in the interval between the development of the two sets of precision estimates.

AASHTO Standard Method of Test T 104

Six AMRL proficiency data sets that reflect the 1,999 revisions to AASHTO T 104 were analyzed to derive precision estimates. The majority of the data sets represent test results from greater than 100 laboratories. The data sets were selected to include a wide range of sodium and magnesium sulfate soundness values.

Criteria developed in this research for judging the acceptability of percentage loss of coarse and fine aggregates by the sulfate soundness test (AASHTO T 104) are given in Table 4. The figures in the second column of Table 4 are the coefficients of variation that have been found to be appropriate for the materials and conditions of test described in the first column of the table. The figures in the third column of the table are the limits that should not be exceeded by the difference between the results of two properly conducted single-operator or multi-laboratory tests as a percent of their mean. The bias of AASHTO T 104 was not determined because no comparison

Table 3 Comparison of the new and existing precision estimates for AASHTO T 22

	Coefficient of Variation, Percent of Mean		Acceptable Range of Two Test Results, Percent of Mean	
Condition of Test and Type Index	New	Existing	New	Existing
Single-Operator Precision:				
6 × 12 in. (150 × 300 mm)	4.7	2.4	13.1	6.6
4 × 8 in. (100 × 200 mm)	4.3	3.2	12.0	9.0
Multi-Laboratory Precision:				
6 × 12 in. (150 × 300 mm)	8.1	5.0	22.8	14.0
4 × 8 in. (100 × 200 mm)	7.6	NA	21.4	NA

Table 4 Precision estimates for AASHTO T 104

Material and Type Index	Coefficient of Variation (1s), Percent of Mean*	Difference Between Two Tests (d2s), Percent of Mean*
Single-Operator Precision:		
Coarse Aggregate		
Sodium Sulfate	19	53
Magnesium Sulfate	18	51
Fine Aggregate		
Sodium Sulfate	12	35
Magnesium Sulfate	10	27
Multi-Laboratory Precision:		
Coarse Aggregate		
Sodium Sulfate	68	190
Magnesium Sulfate	60	168
Fine Aggregate		
Sodium Sulfate	52	145
Magnesium Sulfate	45	125

*These values represent the 1s and d2s limits described in ASTM Practice C670.

NOTE: The precision estimates given in Table 4 are based on an analysis of the weighted average of sulfate soundness loss test results from 60 pairs of AMRL proficiency samples. The data analyzed consisted of results from 90 to 282 laboratories for each of the pairs of samples. The analysis of coarse aggregate sulfate soundness included 19.0-mm to 4.75-mm aggregate with a weighted average sodium sulfate loss of 0.3% to 3.5% and an average magnesium sulfate loss of 0.4% to 14.9%. The analysis of fine aggregate sulfate soundness included 1.18-mm to 300-micron fine aggregate with an average sodium sulfate loss of 1.7% to 2.2% and an average magnesium sulfate loss of 3.3% to 5.21%.

with a material having an accepted reference value was conducted.

The new and existing precision estimates for AASHTO T 104-99 (2003) are provided in Table 5; the single operator precision of the sodium sulfate soundness procedure was reduced while all other precisions increased in comparison to the existing precisions. However, the repeatability and reproducibility coefficient of variations of both new and existing precisions are so large that their utility for within- and between-laboratory comparisons are uncertain. Improving the precisions of the test will require comprehensive ruggedness testing that examines the key variables of the test method.

AASHTO Standard Method of Test T 105

Ten AMRL proficiency data sets were analyzed to derive the precision estimates. The majority of the data sets represent test results from more than 100 laboratories. The data sets were selected to include Type I and Type I/II cements with and without limestone and Type V cement with limestone.

Criteria for judging the acceptability of percentages of chemical components that are obtained using AASHTO T 105 for hydraulic cement are presented in Table 6. The figures in the second column of Table 2 are the standard deviations that were found to be appropriate for the chemical components in the first column of the table. 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 the third column of Table 6. The figures in the fourth column of the table are the multi-laboratory standard deviations that have been found to be appropriate for the chemical components in the first column. Two results submitted by two different operators testing the same material in different laboratories should not be considered suspect unless the difference in the two results exceeds the values given in the fifth column. The bias of AASHTO T 105 could not be determined because no comparison with a material having accepted reference values was conducted.

A comparison of the new and existing precision estimates for AASHTO T 105-06 showed a significant improvement in the precision of several of the chemical analyses. This improvement likely results

Table 5 Comparison of the new and existing T 104 precision estimates

Material and Type Index	New Precisions: Coefficient of Variation (1s), Percent	New Precisions: Difference Between Two Tests (d2s), Percent of Average	Existing Precisions (AASHTO T104-99(2003)): Coefficient of Variation (1s), Percent	Existing Precisions (AASHTO T104-99(2003)): Difference Between Two Tests (d2s), Percent of Average
Single-Operator Precision:				
Coarse Aggregate				
Sodium Sulfate	19	53	24	68
Magnesium Sulfate	18	51	11	31
Fine Aggregate				
Sodium Sulfate	12	35	–	–
Magnesium Sulfate	10	27	–	–
Multi-Laboratory Precision:				
Coarse Aggregate				
Sodium Sulfate	68	190	41	116
Magnesium Sulfate	60	168	25	71
Fine Aggregate				
Sodium Sulfate	52	145	–	–
Magnesium Sulfate	45	125	–	–

Table 6 Precision estimates for AASHTO T 105

Chemical Components	Single-Operator Precision: Standard Deviation (1s)*	Single-Operator Precision: Acceptable Range of Two Test Results (d2s)*	Multi-Laboratory Precision: Standard Deviation (1s)*	Multi-Laboratory Precision: Acceptable Range of Two Test Results (d2s)*
SiO_2 (silicon dioxide)	0.119	0.333	0.196	0.549
Al_2O_3 (aluminum oxide)	0.073	0.204	0.110	0.308
Fe_2O_3 (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_3 (sulfur trioxide)	0.047	0.132	0.076	0.213
LOI (loss on ignition)	0.055	0.154	0.085	0.238
Na_2O (sodium oxide)	0.013	0.036	0.024	0.067
K_2O (potassium oxide)	0.009	0.025	0.016	0.045
TiO_2 (titanium dioxide)	0.005	0.014	0.007	0.020
Cl (chloride)	0.002	0.006	0.004	0.011
IR (insoluble residue)	0.048	0.134	0.080	0.224
Free calcium oxide	0.125	0.350	0.214	0.599
CO_2 (carbon dioxide)	0.083	0.232	0.219	0.613

* These values represent the 1s and d2s limits described in ASTM Practice C670.

NOTE: The precision estimates given in Table 6 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.

from recent advancements in methods for chemical analysis of hydraulic cement.

AASHTO Standard Method of Test T 154

The Gillmore initial and final times of setting of hydraulic cement were analyzed for each of six cement paste proficiency sample pairs. Precision estimates are based, where appropriate, on either the coefficients of variation (CV%) or the pooled standard deviation (1s) values.

Criteria for judging the acceptability of Gillmore initial and final times of setting obtained by AASHTO T 154 are given in Table 7. The figures given in the second column of Table 7 are the standard deviations that have been found to be appropriate for the materials and conditions of the test described in the first column of the table. The figures in the third column of the table are the limits that should not be exceeded by the difference between the results of two properly conducted single-operator or multi-laboratory tests. The bias of AASHTO T 154 was not determined because no comparison with a material having an accepted reference value was conducted.

Table 8 compares the new and existing precision estimates for AASHTO T 154; both repeatability and reproducibility statistics are improved, likely due to recent improvements to the test method.

AASHTO Standard Method of Test T 186

The early stiffening of hydraulic cement measured with the Vicat apparatus was analyzed for each of six cement paste proficiency sample pairs. Precision estimates are based, where appropriate, on either the CV% or the pooled 1s values.

Table 7 Precision estimates of time of setting of hydraulic cement paste by Gillmore needles

Condition of Test and Test Property	Standard Deviation (1s), Minutes*	Acceptable Range of Two Test Results (d2s), Minutes*
Single-Operator Precision:		
Initial Time of Setting	12	34
Final Time of Setting	16	46
Multi-Laboratory Precision:		
Initial Time of Setting	23	64
Final Time of Setting	37	103

* These values represent the 1s and d2s limits described in ASTM Practice C670.

NOTE: The precision estimates given in Table 7 are based on the analysis of test results from 6 pairs of CCRL proficiency samples. The data analyzed consisted of results from 156 to 168 laboratories for each of the pairs of samples. The analysis included cement pastes with an average Gillmore initial time of setting of 139 to 193 min and an average Gillmore final time of setting of 238 to 303 minutes.

Criteria are given in Table 9 for judging the acceptability of early stiffening of hydraulic cement paste by AASHTO T 186 and expressed as the ratio of the final to initial penetration calculated as a percentage. The figures given in the second column of Table 9 are the standard deviations that have been found to be appropriate for the materials and conditions of the test described in the first column of the table. The figures in the third column of Table 9 are the limits that should not be exceeded by the difference between the results of two properly conducted

Table 8 Comparison of new and existing precision estimates for AASHTO T 154

Condition of Test and Type Index	Standard Deviation (1s), Minutes		Acceptable Range of Two Test Results (d2s), Minutes	
	New	Existing	New	Existing
Single-Operator Precision:				
Initial Time of Setting	12	16	34	44
Final Time of Setting	16	22	46	62
Multi-Laboratory Precision:				
Initial Time of Setting	22	28	63	78
Final Time of Setting	37	46	104	129

Table 9 Precision estimates of early stiffening of hydraulic cement (paste method)

Condition of Test and Test Property	Standard Deviation (1s), Percent*	Acceptable Range of Two Test Results (d2s), Percent*
Single-Operator Precision	7	21
Multi-Laboratory Precision	8	24

*These values represent the 1s and d2s limits described in ASTM Practice C670.

NOTE: The precision estimates given in Table 9 are based on the analysis of test results from 6 pairs of CCRL proficiency samples. The data analyzed consisted of results from 171 to 210 laboratories for each of the pairs of samples. The analysis included cement pastes with average False Set of 69% to 85%.

single-operator or multi-laboratory tests. The bias of the procedure was not determined because no comparison with a material having an accepted reference value was conducted.

Table 10 compares the new and existing precision estimates for AASHTO T 186; both repeatability and reproducibility statistics have improved, likely due to the recent improvements in the test method.

AASHTO Standard Method of Test T 242

The precision statement for AASHTO T 242 is based on repeatability and reproducibility standard deviations of the final state friction system measurements.

The single operator and multi-operator standard deviations (1s limits) for FN (friction number unit) are shown in the second column of Table 11. The results of two properly conducted friction tests on the same surface, by the same operator, and using the same equipment, should be considered suspect if they differ by more than d2s single operator limits shown in the third column of Table 11. The results of two properly conducted tests on the same surface, by different operators, using different systems, should be considered suspect if they differ by more than the d2s multi-laboratory limit shown in the third column of Table 11. No information can be presented on the bias of the procedure because no material having an accepted reference value was available.

The current version of AASHTO T 242 includes a repeatability standard deviation that can be compared with the repeatability standard deviation computed in this study. The repeatability standard deviation is reported as 2 FN in AASHTO T 242-96 (2004), which is significantly larger than the computed repeatability standard deviation of 0.83 FN computed in this study. This difference is likely due to careful calibration of state friction systems during their evaluation at TTI and TRC.

Table 10 Comparison of the recommended and existing precision estimates for AASHTO T 186

Condition of Test and Type Index	Standard Deviation (1s), Percent		Acceptable Range of Two Test Results (d2s), Percent	
	New	Existing	New	Existing
Single-Operator Precision	7	10	21	28
Multi-Laboratory Precision	8	12	24	34

Table 11 Precision estimates of friction number

Condition of Test and Test Property	Standard Deviation (1s)*	Acceptable Range of Two Test Results (d2s), Percent
Single-Operator Precision	0.83	2.35
Multi-Laboratory Precision	1.90	5.37

*These limits are determined from data obtained during the calibration and testing of state friction systems at the field test centers at the Texas Transportation Institute and the Transportation Research Center.

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