Interlaboratory Study to Establish Precision Statements for AASHTOT 358 and AASHTOT 402, Electrical Resistivity of Cylindrical Concrete Specimens

Project Report March 2024

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INTERLABORATORY STUDY TO ESTABLISH PRECISION STATEMENTS FOR AASHTO T 358 AND AASHTO T 402, ELECTRICAL RESISTIVITY OF CYLINDRICAL CONCRETE SPECIMENS

Project Report March 2024

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1. INTRODUCTION

An interlaboratory study (ILS) was conducted to establish a precision and bias statement for AASHTO T 358-22, Surface Resistivity Indication of Concrete's Ability to Resist Chloride Ion Penetration, and AASHTO T 402-23, Electrical Resistivity of a Concrete Cylinder Tested in a Uniaxial Resistance Test.

According to the American Association of State Highway and Transportation Officials (AASHTO) Committee on Materials and Pavements (COMP) Information and Operations Guide (June 2018), "Guidance for conducting round-robin testing programs to develop precision is in ASTM C802, C670, E177 and E691."

The ILS was performed by 18 different laboratories for AASHTO T 358 testing and by 9 different laboratories for AASHTO T 402 testing. Personnel performing the testing demonstrated experience with the test method under consideration to ensure that they were competent in running the test. Since all testing was conducted in different laboratories by operators using their own equipment, the ILS was considered a multi-operator, multiple-set-of-apparatus scenario.

The overall study was divided into five phases: A through E. Phase A involved identifying participating laboratories. Phase B involved developing and delivering training tools. Phase C involved determining single-operator variability. Phase D involved determining multi-laboratory variability. Phase E involved preparation of the final report.

In Phase A, a survey created using Google Forms was distributed that could be submitted by any laboratory that was interested in participating in the ILS. Details regarding the resistivity equipment used by the laboratories were also collected through the survey. Phase B involved developing and delivering training tools for the participating laboratories, such as recorded webinars on how to use the verification device and run the resistivity test and data entry spreadsheet templates. Phase C involved collecting the data required to determine single-operator and multi-laboratory variability in measurements made using the verification device. In addition, Phase C involved preparing and conditioning concrete samples at Oregon State University that were then used to determine the single-operator variability in resistivity measurements of the concrete samples. Phase D involved preparing concrete samples and shipping them to the participating laboratories, where the samples were then conditioned and measured for resistivity. The data from Phase D were used to determine the multi-laboratory variability in resistivity measurements of the concrete samples. The concrete samples tested in Phase C were made from two different mixtures (one designed for high resistivity and one designed for low resistivity), and the concrete samples tested in Phase D were similarly made from two different mixtures (again, one designed for high resistivity and one designed for low resistivity). The same mixture proportions were used across both phases.

2. TEST METHODS

The test methods used for this ILS were AASHTO T 358-22 and AASHTO T 402-23. To obtain a copy of AASHTO T 395 and AASHTO T 402, go to the AASHTO store at https://store.transportation.org/ or contact AASHTO by phone at (800) 231-3475.

3. PARTICIPATING LABORATORIES AND OPERATORS

The following laboratories provided operators for this interlaboratory study: S.T.A.T.E. Testing LLC, Texas Department of Transportation (DOT), Ash Grove Cement, Federal Highway Administration (FHWA) Mobile Concrete Technology Center, New York DOT, Massachusetts DOT, Idaho DOT, Tennessee DOT, Kansas DOT, Wyoming DOT, West Virginia DOT, Braun Intertec, Utah DOT, Kansas DOT, Vermont Agency of Transportation, Connecticut DOT, Oregon DOT, FHWA Turner-Fairbank Highway Research Center (TFHRC), and Oregon State University. See Appendix A for a listing of participants.

4. DESCRIPTION OF MIXTURES AND SAMPLES

Two mixtures each were used in Phase C and Phase D of the study, with the same mixture proportions used in both phases. Mixture 1 was designed to have a low electrical resistivity corresponding to high chloride penetration, and Mixture 2 was designed to have a high electrical resistivity corresponding to low chloride penetration. Mixture 1 was a 100% ordinary portland cement (OPC) mixture with a total cementitious content of 500 lb/yd³ and a design water-to-cementitious material (w/cm) ratio of 0.52. Mixture 2 was a modified high-performance concrete mixture with a total cementitious content of 750 lb/yd³, including 30% slag and 4% silica fume, and a design w/cm ratio of 0.37. No air-entraining admixtures were used in either of the mixtures. Coarse aggregate (¾ in.) and concrete sand were used for both of the mixtures (in compliance with ASTM C33). The concrete mixtures were prepared by Knife River Corporation of Corvallis, Oregon.

In both Phase C and Phase D, the batch for each mixture consisted of 5 yd³ of concrete, and the first one-third of the concrete from each ready-mix truck was discarded before the rest was used for making the samples. The concrete cylinders, each having a diameter of 4 in. and a length of 8 in., were made at the concrete plant. For Phase C, all of the samples were cured and conditioned at Oregon State University. For Phase D, the samples were cast, demolded within 24 hours of casting, and shipped in sealed conditions within three days after demolding to all of the participating laboratories.

AASHTO T 358 involves conditioning and curing samples in saturated lime solution, whereas AASHTO T 402 provides two conditioning options: Option A, conditioning in simulated pore solution, and Option B, sealed conditioning. For each conditioning method, three concrete cylinders were prepared for each laboratory. The samples for Phase C were tested at an age of 42 days, and the samples for Phase D were tested at an age of 56 days.

5. INTERLABORATORY STUDY INSTRUCTIONS

Prior to the ILS, laboratory participants were provided with copies of AASHTO T 358-22 and AASHTO T 402-23 and were emailed detailed test program instructions, including short illustrative videos explaining how to perform the test and use the verification device. Additionally, the technical team conducted short webinars before each phase of testing that included instructions on conditioning and testing the samples and collecting and reporting the data. Presentation titles included "Resistivity—Precision and Bias," "Instructions for Phase D Sample Conditioning," and "Instructions for Phase D Sample Measurements."

6. DESCRIPTION OF EQUIPMENT/APPARATUS

The equipment listed in this section and in Appendix B was used to develop a precision statement for AASHTO T 358-22 and T 402-23. This listing is not an endorsement or certification by AASHTO.

Each participating laboratory received a verification device to be used for bias testing. Designed prior to this study by S. Chopperla, O. B. Isgor, and W. J. Weiss, the verification device consists of an acetal resin bar with two stainless steel electrodes (each 4 in. in diameter) attached to its ends. It also includes a row of four screws spaced 38 mm (1.49 in.) apart on one of its sides for surface configuration measurements. The total length of the verification device is 8 in. to replicate the dimensions of the standard 4 in. x 8 in. (101.6 mm x 203.2 mm) concrete cylinder. The steel end plates and the four screws on the side are connected to an electrical circuit. The circuit is designed such that the operators can choose either a bulk or surface configuration and either a low- or high-impedance circuit.

The resistor-capacitor (RC) circuits used for the verification device are diagrammed in Appendix B. The impedance values of the designed low- and high-impedance circuits are 610 ohms and 5210 ohms, respectively. The resistivities corresponding to the low- and high-impedance circuits for a 4 in. x 8 in. cylinder configuration are 24.3 ohm.m and 207.9 ohm.m, respectively. These resistivity values correspond to high and very low chloride penetration levels in concrete (as defined in ASTM C1202), thus covering a wide range of resistivity values.

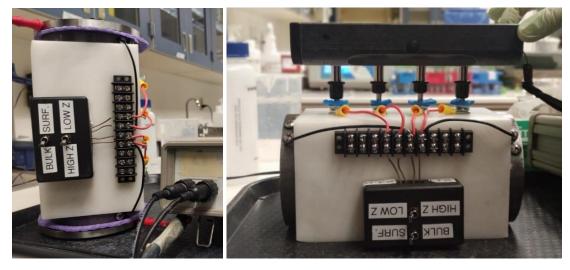


Figure 1. Verification device in bulk and surface configurations

The resistivity equipment used for this study is described in AASHTO T 402 and summarized in Appendix B.

7. DATA REPORT FORMS

Each ILS operator was provided with a data entry template to record test results. A copy of the test results is provided in Appendix C.

Note: The ILS operators have been randomly coded and are not identified with specific testing stations herein.

8. STATISTICAL DATA SUMMARY

Both standards specify testing at least two samples; for this study, three samples were tested for each conditioning method. The coefficient of variation was determined and used to develop the single-operator and multi-laboratory data for both the verification device and the concrete samples tested in accordance with AASHTO T 358 and AASHTO T 402, as shown in Tables 1 and 2, respectively. The minimum acceptable difference between two operators' results was determined as described in ASTM C 670-15 using the d2s value of 2.8; these values are also given in Tables 1 and 2. All collected data were included in the calculations, and no outliers were found.

9. PRECISION AND BIAS STATEMENT

9.1. Precision

The single-operator and multi-laboratory precision results are described as follows.

Single-Operator Precision—The coefficient of variation is used to present the single-operator precision, as shown in Table 1.

Multi-Laboratory Precision—The coefficient of variation is also used to present the multi-laboratory precision, as shown in Table 2.

Table 1. Indices of precision for resistivity determined according to AASHTO T 358

Preci	sion Indices	Coefficient of Variation (%)	Maximum Acceptable Difference between Two Operators' Results, d2s (%)
Verification	Single-Operator	0.2	0.4
Device	Multi-Laboratory	0.5	1.3
Concrete	Single-Operator	5.8	16.1
Specimens	Multi-Laboratory	10.9	30.5

Table 2. Indices of precision for resistivity determined according to AASHTO T 402

			Maximum Acceptable
		Coefficient of	Difference between Two
Precision Indices		Variation (%)	Operators Results, d2s (%)
Verification Device	Single-Operator	0.2	0.5
verification Device	Multi-Laboratory	2.1	5.8
Concrete Specimens,	Single-Operator	3.3	9.2
Conditioning Option A ¹	Multi-Laboratory	13.0	36.5
Concrete Specimens,	Single-Operator	3.4	9.5
Conditioning Option B ²	Multi-Laboratory	11.3	31.7

¹Conditioning in pore solution

9.2. Bias

The verification device was used as a reference material for determining the bias of the test methods. The bias of the AASHTO T 358 test method was found with 95% confidence to lie between -1.8% and -2.0%. The bias of the AASHTO T 402 test method was found with 95% confidence to lie between 2.0% and 2.4%.

² Sealed sample conditioning

APPENDIX A. PARTICIPANTS (INCLUDING BOTH TESTERS/OPERATORS AND OBSERVERS)

Table A-1. Participants

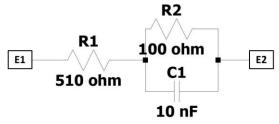
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APPENDIX B. DESCRIPTION OF EQUIPMENT/APPARATUS

Surface Resistivity Apparatus—Wenner probes provided by two different suppliers were used in this study to measure surface resistivity. Each device had a probe tip spacing of 38.1 mm (1.5 in.) and was capable of supplying a frequency between 10 and 1,000 Hz.

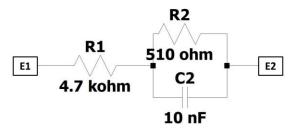
Bulk Resistivity Apparatus—Two types of devices were used in this study to measure bulk resistivity. One was an alternating current resistivity meter capable of supplying a frequency between 10 and 1,000 Hz, an example of which is shown in Figure 2b of AASHTO T 402-23. The other was a surface resistivity meter that can be used with a modified configuration, as described in Section 11.3.1 and shown in Figure 2c of AASHTO T 402-23.

Figures B-1 through B-4 show the circuits used for the verification device. More details on the design of the verification device are available in an in-progress paper by K. S. T. Chopperla, A. L. de Sequeira Neto, O. B. Isgor, and W. J. Weiss entitled "Measuring Electrical Resistivity of Concrete Cylinders: Precision and Bias."



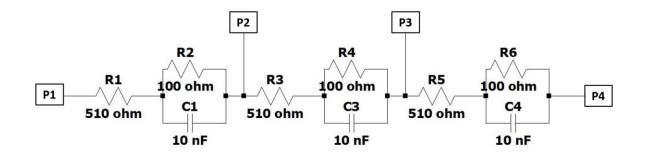
E1 and E2 represent the electrodes used in the bulk configuration.

Figure B-1. Low-impedance circuit for the bulk configuration



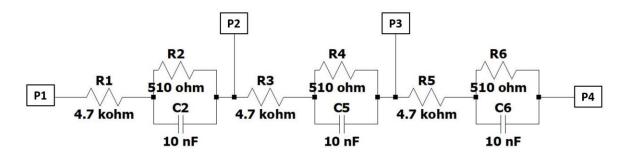
E1 and E2 represent the electrodes used in the bulk configuration.

Figure B-2. High-impedance circuit for the bulk configuration



P1, P2, P3, and P4 represent the four probes of the surface resistivity device.

Figure B-3. Low-impedance circuit for the surface configuration



P1, P2, P3, and P4 represent the four probes of the surface resistivity device.

Figure B-4. High-impedance circuit for the surface configuration

APPENDIX C. RAW DATA

Table C-1 provides the resistance measurements calculated from the low- and high-impedance circuits of 18 verification devices operated by individual operators.

Table C-1. Single-operator data for the verification devices

	Resistance, Ω (Surface Configuration)			ance, Ω figuration)
S.	High-Impedance	Low-Impedance	High-Impedance	Low-Impedance
no.	Circuit	Circuit	Circuit	Circuit
1	5,105.5	596.8	5,314.4	622.4
2	5,105.5	598.9	5,314.4	625.3
3	5,105.5	596.8	5,314.4	622.4
4	5,105.5	596.8	5,314.4	623.3
5	5,101.3	598.9	5,315.3	624.3
6	5,105.5	598.9	5,315.3	624.3
7	5,105.5	598.9	5,315.3	624.2
8	5,105.5	598.9	5,315.3	624.3
9	5,105.5	598.9	5,311.1	622.1
10	5,105.5	598.9	5,316.1	624.1
11	5,105.5	598.9	5,306.1	623.1
12	5,105.5	598.9	5,306.1	623.0
13	5,105.5	594.7	5,316.1	626.0
14	5,105.5	598.9	5,316.1	625.0
15	5,105.5	598.9	5,311.1	623.6
16	5,105.5	598.9	5,316.1	622.1
17	5,101.3	596.8	5,316.1	627.0
18	5,103.4	598.9	5,317.5	621.0

From the data in Table C-1, the single-operator precision of the verification device and the respective biases for AASHTO T 358 and AASHTO T 402 were determined. For the surface configuration, the upper limits for the bias were found with 95% confidence to be -2.0% and -1.8% for AASHTO T 358 and AASHTO T 402, respectively. For the bulk configuration, the upper limits for the bias were found with 95% confidence to be 2.0% and 2.4% for AASHTO T 358 and AASHTO T 402, respectively.

Table C-2 provides the resistance measurements calculated from the low- and high-impedance circuits of the verification devices operated by the participating laboratories.

Table C-2. Multi-laboratory data for the verification devices

	Resist	ance, Ω	Resistance, Ω		
	(Surface Co	onfiguration)	(Bulk Configuration)		
S.	High-Impedance	Low-Impedance	High-Impedance	Low-Impedance	
no.	Circuit	Circuit	Circuit	Circuit	
1	5,109.7	598.9	5,220.0	632.0	
2	5,101.3	598.9	5,312.8	640.8	
3	5,105.5	598.9	5,308.6	620.6	
4	5,113.9	594.7	5,318.0	633.0	
5	5,105.5	598.9	5,164.2	661.7	
6	5,197.7	607.3	5,155.8	645.0	
7	5,105.5	598.9	5,180.9	611.5	
8	5,105.5	598.9	5,113.9	607.3	
9	5,105.5	598.9	5,160.0	645.0	
10	5,101.3	598.9	5,134.8	615.7	
11	5,101.3	598.9	5,118.1	607.3	
12	5,109.7	598.9	5,113.9	603.1	
13	5,105.5	598.9	~	~	
14	5,101.3	594.7	~	~	

Tables C-3 and C-4 provide average resistivity values determined following AASHTO T 358 and AASHTO T 402 that were used to calculate the single-operator and multi-laboratory variation. Normalized resistivity values were calculated for each mixture by dividing the resistivity values of each test with the average resistivity value determined from all of the tests. The coefficient of variation (COV) was then calculated by determining the standard deviation of all of the normalized values for both of the mixtures.

Table C-3. Resistivity data collected for calculating single-operator precision (Phase C)

	Resistivity, kΩ-cm (AASHTO T 358)		Resistivity, kΩ-cm (AASHTO T 402, Option A)		Resistivity, kΩ-cm (AASHTO T 402, Option B)	
Test	Mixture 1	Mixture 2	Mixture 1	Mixture 2	Mixture 1	Mixture 2
1	2.90	8.52	1.86	6.19	3.31	10.82
2	2.66	8.30	1.87	6.73	3.20	9.94
3	2.65	8.01	1.88	6.34	3.13	10.36
4	2.78	8.38	1.88	6.30	3.48	10.15
5	2.69	8.51	1.81	6.68	3.21	10.34
6	2.74	8.43	1.82	6.41	3.55	10.53
7	2.92	8.10	1.73	6.53	3.46	10.13
8	2.80	8.14	1.83	6.69	3.34	10.66
9	2.70	7.04	1.69	6.80	3.40	10.67
10	3.24	8.47	1.82	6.47	3.27	10.69
COV	5.8	3%	3.3	3%	3.4	1%

Table C-4. Resistivity data collected for calculating multi-laboratory precision (Phase D)

		y, kΩ-cm	Resistivity, kΩ-cm		Resistivity, kΩ-cm	
	(AASHTO T 358)		(AASHTO T 402, Option A)		(AASHTO T 402, Option B)	
Test	Mixture 1	Mixture 2	Mixture 1	Mixture 2	Mixture 1	Mixture 2
1	2.78	10.11	1.95	9.70	2.84	12.32
2	3.01	13.62	1.98	10.59	3.17	15.30
3	2.68	12.20	2.09	11.61	3.11	15.17
4	3.15	11.87	2.16	10.33	3.65	15.22
5	2.83	10.76	2.22	10.44	3.12	13.31
6	3.03	9.65	1.76	8.27	2.74	10.37
7	2.83	11.40	1.94	13.26	3.43	14.97
8	2.49	11.53	2.27	8.73	3.21	11.75
9	3.07	11.87	2.20	8.80	3.22	11.93
10	2.78	9.59*	2.30	13.31	3.66	15.02
11	3.14	12.68*	2.06	11.75	3.84	15.20
12	3.23	12.74*	2.13	8.15	3.12	12.71
13	3.03	9.64*	~	~	~	~
14	3.25	12.37	~	~	~	~
15	2.66	11.32*	~	~	~	~
16	2.72	9.31*	~	~	~	~
17	3.28	13.10*	~	~	~	~
18	2.96	9.87	~	~	~	~
COV	10.	9%	13.0%		11.3%	

^{*}Average of resistivity values determined from two samples.

