Interlaboratory Study to Establish a Multi-Laboratory Precision **Statement for AASHTOT 395-22, Characterization of the Air-Void System of Freshly Mixed Concrete** by the Sequential Pressure Method

Project Report September 2023

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INTERLABORATORY STUDY TO ESTABLISH A MULTI-LABORATORY PRECISION STATEMENT FOR AASHTO T 395-22, CHARACTERIZATION OF THE AIR-VOID SYSTEM OF FRESHLY MIXED CONCRETE BY THE SEQUENTIAL PRESSURE METHOD

Project Report September 2023

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Sponsored by Federal Highway Administration (Part of Cooperative Agreement 693JJ31950004, Advancing Concrete Pavement Technology Solutions)

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The concrete supplier for this project was Concrete Supply, Inc. (CSI), Delaware Plant #7, 4000 Delaware Ave, Des Moines, Iowa. The authors would like to thank the staff at CSI, especially Ready Mix Quality Control Manager Jeffrey Goss, for hosting the interlaboratory study at CSI's central laboratory and for their help in organizing the testing.

1. INTRODUCTION

An interlaboratory study (ILS) was conducted to establish a multi-laboratory precision statement for AASHTO T 395-22, Characterization of the Air-Void System of Freshly Mixed Concrete by the Sequential Pressure Method.

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 over 2 days by 14 different operators in one location. Personnel performing the testing reported having previous experience with the test method.

The ILS was considered a multi-laboratory precision assessment since all testing was conducted by different operators using different equipment, despite testing taking place at one location.

2. TEST METHOD

The test method used for this ILS was AASHTO T 395-22. To obtain a copy of AASHTO T 395 go to the AASHTO store at <u>https://store.transportation.org/</u> or contact AASHTO by phone at (800) 231-3475.

3. PARTICIPATING LABORATORIES AND OPERATOR BACKGROUNDS

The following laboratories provided operators for this interlaboratory study: Iowa Department of Transportation (DOT), Kansas DOT Bureau of Research, North Dakota DOT, Oklahoma State University, American Engineering Testing, GCP Applied Technologies, Braun Intertec, Michigan Concrete Association, Concrete Supply, Inc., and FHWA. The National Concrete Pavement Technology Center (CP Tech Center) invited industry stakeholders to attend, and personnel from FHWA and the Iowa Concrete Paving Association also attended. See Appendix A for a listing of participants and Appendix B for the user-reported operator survey information.

4. DESCRIPTION OF SAMPLES

The base concrete mixture was a Class C-SUD mixture that is commonly used by Iowa municipalities for concrete paving; the mixture features an optimized gradation with intermediate aggregate and 20% Class C fly ash replacement. The mixture proportions are provided in Appendix C. Each 5 yd³ concrete batch was proportioned with a target slump of 2 to 2.5 in. Air contents were adjusted for each concrete batch to ensure that the experiment included air contents between 3% and 8%.

The ILS included four 5 yd³ concrete batches (batches 1, 2, 3, and 4) with varying target air contents. Each batch was tested twice, with runs designated A and B. The runs constituted two separate samples due to the time between removal from the truck and the addition of mixture water to the approximately 60% of the concrete load that remained. The concrete mixtures were prepared and delivered to the testing location by Concrete Supply, Inc. (CSI). CSI's production plant was located approximately 2 miles away from the testing location, CSI's central laboratory.

5. INTERLABORATORY STUDY INSTRUCTIONS

Prior to the ILS, laboratory participants were emailed test program instructions and videos discussing how to run the test and calibrate the meter. The day prior to the ILS, operators were given an overview of the method. A copy of the instructions is included in Appendix D.

6. DESCRIPTION OF EQUIPMENT/APPARATUS

The equipment used for this study is described in AASHTO T 395-22 and summarized in Appendix E.

7. DATA REPORT FORMS

Each ILS operator was provided with a data report form to record test results. On this form, the user wrote the equilibrium pressures at each step. These were recorded because they were the fundamental measurements of the test method. The Super Air Meter (SAM) number and air content recorded by the operator were used for the analysis in this report. A copy of the SAM number and air content data is provided in Appendix F. For information on mixture proportions, see Appendix C. For information on other measured properties, see Appendix G.

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

8. STATISTICAL DATA SUMMARY

The standard deviation/coefficient of variation was determined and used to develop the multilaboratory data in Tables 1 and 2. The minimum acceptable difference between two results was determined as described in ASTM C670 using the d2s value of 2.8 multiplied by the standard deviation. (The standard deviation for each air and SAM number range is the product of the average and coefficient of variation.) Data were not eliminated from the air content testing. Some data were omitted from the ILS for the SAM number due to equipment failure, which was determined from a leak check immediately after the test. In addition, two data points were removed from the SAM number because they were outside the allowable range per the standard (0.03 to 0.82). No other data points were removed. Plots of these data are provided in Appendix H and Appendix I.

9. PRECISION AND BIAS STATEMENT

9.1. Precision

Multi-Laboratory Precision— The multi-laboratory statistics have been established for the air content and the SAM Number.

The multi-laboratory standard deviation was found to increase with air content, as shown in Table 1 for air contents within the range of 4% to 8%. Therefore, the results of two properly conducted tests by different laboratories on the same material are not expected to differ from each other by more than the value shown in the last column of Table 1.

For air contents less than 4%, the air contents of two properly conducted tests by different laboratories on the same material are not expected to differ from each other by more than 0.67%.

Note: Precision data can be presented by either standard deviation or coefficient of variation (in this case, a single standard variation of 0.32 or a coefficient of variation of 5.9%). For the purposes of this effort, it was decided to use the coefficient of variation for consistency with ASTM C231.

The multi-laboratory standard deviation was found to increase with SAM number, as shown in Table 2 for SAM numbers within the range of 0.10 to 0.40. Therefore, the results of two properly conducted tests by different laboratories on the same material are not expected to differ from each other by more than the value shown in the last column of Table 2.

For SAM numbers less than 0.1, the SAM numbers of two properly conducted tests by different laboratories on the same material are not expected to differ from each other by more than 0.113.

Table 1. Multi-laboratory	indexes of precision	for air contents	(volume) between	1 4% and
8% ^{A, B}				

		Acceptable Difference
Air Content (%)	Standard Deviation (%)	between Two Results ^C %
4	0.24	0.67
5	0.30	0.84
6	0.35	0.98
7	0.41	1.15
8	0.47	1.32

^A Use interpolation to determine precision values for air contents between the values given in the table.

^B The coefficient of variation of a single test for the volume of air was found to be 5.9%.

^c These numbers represent the d2s limits as prescribed in ASTM C670.

SAM Number	Standard Deviation	Acceptable Difference between Two Results ^C %
0.10	0.040	0.113
0.15	0.060	0.169
0.20	0.081	0.226
0.25	0.101	0.282
0.30	0.121	0.339
0.35	0.141	0.395
0.40	0.161	0.451

Table 2. Multi-laboratory indexes of precision for average SAM values between 0.1 and 0.4 $_{\rm A,\,B}$

^A Use interpolation to determine precision values for SAM numbers between the values given in the table. ^B The coefficient of variation is 40.3%.

^c These numbers represent the d2s limits as prescribed in ASTM C670.

9.2. Bias

At the time of the study, there was no accepted reference material suitable for determining the bias for this test method. Therefore, no statement on bias is made herein.

9.3. Methods

The precision statement was determined through statistical examination of 106 air content tests and 93 SAM results obtained by 14 operators on 8 samples. The precision of this test method is based on an interlaboratory study of AASHTO T 395-22 conducted in March 2023. Every "test result" represents an individual determination. ASTM E691 was followed for the design and analysis of the data.

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

Table A-1. Participants

		Tester (T) or
Organization	Name	Observer (O)
Iowa DOT (Slump, Air, and UW, not SAM)	Todd Hanson	Т
Iowa DOT	Christian Barko	Т
Iowa DOT	Joe Burns	Т
KDOT Bureau of Research	Dan Wadley	Т
KDOT Bureau of Research	Sally Mayer	Т
North Dakota DOT	Terry Fuchs	Т
North Dakota DOT	Justin Rogstad	Т
Oklahoma State University	Jake LeFlore	Т
Oklahoma State University	Bahaa Abdelrahman	Т
American Engineering Testing	Devin Quinn	Т
GCP Applied Technologies	Ryan Scott	Т
Braun Intertec	John Pomranke	Т
Michigan Concrete Association	Steve Waalkes	Т
Concrete Supply, Inc.	Cassandra Goss	Т
FHWA	Nikolai Morari	Т
Concrete Supply, Inc.	Jim Viers	0
Concrete Supply, Inc.	Jeff Goss	0
CP Tech Center	Peter Taylor	0
CP Tech Center	Leif Wathne	0
CP Tech Center	Dan King	0
CP Tech Center	Peter Taylor	0
Oregon State University	Jason Weiss	0
Oklahoma State University	Tyler Ley	0
Mark E. Felag, LLC	Mark Felag	0
FHWA	Jagan Gudimettla	0
Iowa Concrete Paving Association	Greg Mulder	0

APPENDIX B. SAM ROUND-ROBIN OPERATOR SURVEY

Lab/Test	Question 1	Question 2	Question 3	Question 4	Question 5
1	Experienced user	Monthly	~ Dr. Ley, YouTube		Research
2	Somewhat familiar	Every 6 months	YouTube	~	Bridge decks/paving
3			No operator		
4	Experienced user	Weekly	Dr. Ley	SAM class DOT	Field testing
5	Experienced user	Every 6 months	SAM class DOT	PCA training	Research projects
6			No operator		
7	Experienced user	Monthly	Developed SAM with Dr. Ley Trained several DOTs		Multifunctional
8	Regular user	Daily	~	Video hands-on	Informational
9	Somewhat familiar	Every 6 months	DOT training; YouTube	~	Lab
10	Somewhat familiar	Every 6 months	YouTube; AASHTO	~	Field testing
11	Somewhat familiar	Training and calibrations	Hands-on, YouTube Hands-on at DOT		Field assessment
12	Experienced user	Monthly	~ ~		Tech transfer
13	Experienced user	Monthly	6 years PCA training		Assess air
14	Regular user	Monthly	SAM class DOT	SAM class DOT	Assess lab mixtures
15	Regular user	Every 3 months	SAM at DOT SAM class DOT Traini		Training/certification
16	Experienced user	Every 6 months	FHWA training	FHWA training	Assessment

Table B-1. Results of SAM round-robin operator survey

Question 1: In your opinion, what level of experience do you have with the SAM?

Question 2: Over the last 12 months, how often have you used the SAM to measure concrete?

Question 3: How did you learn to run the SAM?

Question 4: Please list any formal SAM training that you received and who administered the training.

Question 5: What do you use the SAM for in your job?

~ denotes no response

APPENDIX C. ESTIMATED MIXTURE PROPORTIONS FROM BATCH TICKETS AND FRESH PROPERTIES

Descriptor	Unit	1A	1B*	2A	2B*	3A	3B*	4 A	4B *
Coarse Aggregate	lb/yd ³	1396	1396	1396	1396	1392	1392	1396	1396
Intermediate Aggregate	lb/yd ³	412	412	428	428	404	404	412	412
Fine Aggregate	lb/yd ³	1368	1368	1364	1364	1372	1372	1368	1368
Cement	lb/yd ³	463	463	461	461	458	458	443	443
Class C Fly Ash	lb/yd ³	112	112	110	110	110	110	110	110
Mid-Range Water- Reducing Admixture	oz/yd ³	16.6	16.6	16.8	16.8	16.8	16.8	16.4	16.4
Water-Reducing Admixture	oz/yd ³	16.4	16.4	16.4	16.4	16.2	16.2	7.8	7.8
Air Entrainer	oz/yd ³	5.6	5.6	4.0	4.0	7.0	7.0	8.0	8.0
Water	lb/yd ³	195	207	179	187	204	223	212	226
Temperature	F	65	72	66	73	62	64	64	68
Slump	in	0.75	2.75	2.25	2.00	2.00	2.50	2.50	2.75
Air	%	5.0	6.0	4.6	4.0	6.8	6.9	7.6	8.0
Estimate Water/Cementitious	~	0.340	0.359	0.313	0.328	0.359	0.393	0.384	0.409

Table C-1. Estimated mixture proportions from batch tickets and fresh properties

* B Mixtures calculated assuming 40% of the concrete was removed from the truck before water redosing

APPENDIX D. INTERLABORATORY STUDY INSTRUCTIONS

A copy of AASHTO T 395-22 was provided to the participants to follow. An overview of the test program summarized in this appendix was provided for all participants as well.

D.1. Notes about Test Program

- There are 14 testers, each assigned to their own station.
- Every tester will share a wheelbarrow with another tester as shown in Table D-1 and Figure D-1.

Table D-1	Wheelbarrow	assignments
-----------	-------------	-------------

Wheelbarrow	Testing Pair
А	1 & 8
В	2 & 4
С	5 & 10
D	7 & 12
E	13 & 14
F	9 & 15
G	11 & 16



Figure D-1. Wheelbarrow assignments for testing stations

D.2. Flow of Testing

- First concrete truck arrives!
 - Once deemed satisfactory... (uniform, slump, air, UW, T)
- Sampling commences (starting with "A" run).

- Each paired testers' wheelbarrow is filled in turn.
- Three cylinders are cast by CP Tech staff from each wheelbarrow.
- The wheelbarrow is transferred to the SAM testing location for SAM tests.
- Once finished, dispose of concrete and clean all equipment.
- Proceed with "B" tests from the same truck (if applicable).
 - CP Tech staff run slump, air, UW, and T tests and cast three cylinders.
- The order of sampling (via shared wheelbarrow) will change from run to run per our randomized order (Table D-2).

Sampling	Team Order by Truck									
Order	1A	1B	2A	2B	3A	3B	4 A	4B	5A	5B
1	А	С	С	Α	В	D	А	С	В	D
2	В	D	Α	G	С	G	С	F	Е	G
3	С	Α	D	В	D	Е	D	В	C	F
4	D	В	G	F	G	В	F	А	А	С
5	Е	G	Е	Е	Α	С	Е	D	F	В
6	F	F	F	С	E	A	В	G	D	E
7	G	Е	В	D	F	F	G	Е	G	Α

Table D-2. Sampling order

- SAM sampling commences from "B" run.
 - Each paired testers' wheelbarrow is filled in turn.
 - The wheelbarrow is transferred to the SAM testing location for SAM tests.
 - Once finished, dispose of concrete and clean all equipment.
- Proceed with sampling from Trucks 2 and 3 in an identical manner.

Note: We plan to test three trucks (5 yd^3 each) in addition to a practice truck (which may turn into Truck 1 if all goes well) to ensure that we're ready to go. The first truck will have a target air content of 5.5%, the second truck a target air content of 3%, and the third truck a target air content of 8% (all with a target slump of 2.5 to 3 in.).

APPENDIX E. DESCRIPTION OF EQUIPMENT/APPARATUS

Air Meter—A device (Figure E-1) consisting of a measuring bowl and cover assembly. The operational principle of this meter consists of sequentially equalizing known volumes of air in a sealed air chamber, at a series of known pressures, with the unknown volume of air in the concrete sample placed in the measuring bowl. A digital pressure gauge with an accuracy of 0.01 psi (0.07 kPa) shall be used. The digital gauge shall be able to compute and report the air content within 0.1% and the SAM number to 0.01 psi (0.07 kPa). The cover assembly shall be fixed to the measuring bowl with the same uniform pressure that was used during the calibration of the meter.



Figure E-1. Air meter

Measuring Bowl—The measuring bowl shall be essentially cylindrical in shape; made of steel, hard metal, or other hard material not readily attacked by the cement paste; have a minimum diameter equal to 0.75 to 1.25 times the height; and have a capacity of at least 0.20 ft³ (5.7 L). It shall be flanged or otherwise constructed to provide for a pressure-tight fit between the bowl and cover assembly. The interior surfaces of the bowl and surfaces of the rims, flanges, and other component-fitted parts shall be machined smooth. The measuring bowl and cover assembly shall be sufficiently rigid to limit the expansion of the apparatus assembly to no more than 0.1% of air content on the indicator scale.

Cover Assembly—The cover assembly shall be made of steel, hard metal, or other hard material not readily attacked by the cement paste. It shall be flanged or otherwise constructed to provide for a pressure-tight fit between bowl and cover assembly and shall have machined-smooth interior surfaces contoured to provide an air space above the level of the top of the measuring bowl. The cover shall be sufficiently rigid to limit the expansion.

The cover assembly shall be fitted with air valves, air bleeder valves, and petcocks for bleeding off water or through which water may be introduced as necessary for the particular meter design. Suitable means for clamping the cover to the bowl shall be provided to make a pressure-tight seal without entrapping air at the joint between the flanges of the cover and bowl. The clamping method should provide a uniform pressure along the seal that can be verified by the user. A suitable hand pump shall be provided with the cover, either as an attachment or as an accessory.

Standardization Vessel—A measure having an internal volume equal to a percent of the volume of the measuring bowl corresponding to the approximate percent of air in the concrete to be tested; or, if the measure is smaller, it shall be possible to check the standardization of the meter indicator at the approximate percent of air in the concrete to be tested by repeated filling of the measure. When the design of the meter requires placing the standardization vessel within the measuring bowl to check standardization, the measure shall be cylindrical in shape and have an inside depth 1/2 in. (13 mm) less than that of the bowl.

Trowel—A standard brick mason's trowel.

Tamping Rod—A round, straight steel rod, with a $\frac{5}{8} \pm \frac{1}{16}$ in. (16 ± 2 mm) diameter. The length of the tamping rod shall be at least 4 in. (100 mm) greater than the depth of the measure in which rodding is being performed but not greater than 24 in. (600 mm) in overall length. The length tolerance for the tamping rod shall be $\pm \frac{1}{8}$ in. (±4 mm). The rod shall have the tamping end or both ends rounded to a hemispherical tip of the same diameter as the rod.

Mallet—A mallet (with a rubber or rawhide head) weighing approximately 1.25 ± 0.50 lb (0.57 ± 0.23 kg) for use with measures of 0.25 ft³ (14 L) or smaller.

Strike-Off Plate—A flat, rectangular metal plate at least $^{1}/_{4}$ in. (6 mm) thick or a glass or acrylic plate at least $^{1}/_{2}$ in. (12 mm) thick with a length and width at least 2 in. (50 mm) greater than the diameter of the measure with which it is to be used. The edges of the plate shall be straight and smooth within a tolerance of $^{1}/_{16}$ in. (1.5 mm).

APPENDIX F. RAW DATA

Tables F-1 and F-2 provide test results from the air content and SAM number testing following AASHTO T 395-22.

Lab/Test	1A	1B	2A	2B	3 A	3B	4 A	4B		
1	4.7	5.4	5.3	3.9	6.5	7.0	7.0	7.8		
2	4.6	~	4.1	~	6.6	8.1	6.5	7.8		
3	No operator									
4	4.6	5.2	~	3.7	6.5	6.6	6.4	7.5		
5	4.7	5.2	4.2	3.8	6.3	6.5	6.8	7.4		
6	No operator									
7	4.4	4.8	4.4	3.6	6.5	6.3	6.7	7.3		
8	4.9	4.8	4.1	3.9	6.4	6.3	6.6	7.5		
9	4.6	4.9	4.1	~	6.4	~	7.0	7.8		
10	4.8	5.4	4.6	3.9	6.8	6.5	7.0	7.9		
11	4.6	5.7	4.6	4.3	6.9	7.2	7.2	8.2		
12	4.4	5.2	4.1	3.5	6.2	6.4	6.5	7.1		
13	4.4	5.5	4.3	3.9	~	6.7	6.8	7.7		
14	3.7	4.8	4.2	3.2	5.4	6.0	6.6	7.9		
15	4.3	5.2	4.2	3.7	6.2	6.1	6.6	7.1		
16	4.4	5.3	4.5	3.9	6.7	6.9	6.8	7.8		
No. of Labs	14	13	13	12	13	13	14	14		
No. of Replicates	1	1	1	1	1	1	1	1		
Average	4.51	5.18	4.36	3.78	6.42	6.66	6.75	7.63		
Std. Deviation	0.28	0.28	0.32	0.26	0.36	0.53	0.23	0.31		

 Table F-1. Normalized air content (%) for ASTM E691

~ denotes no data for that entry

Average standard deviation 0.32

Lab/Test	1A	1 B	2A	2B	3A	3B	4 A	4B			
1	0.18	0.09	~	0.31	0.15	~	0.17	0.14			
2	0.24	~	0.20	~	0.19	0.10	0.14	0.18			
3	No operator										
4	0.43	0.15	~	0.17	0.05	0.14	0.08	0.10			
5	0.19	0.32	0.21	0.32	0.15	0.12	0.09	0.07			
6	No operator										
7	0.22	0.27	0.43	0.58	0.07	0.12	0.14	0.10			
8	0.11	0.09	0.05	0.20	~	0.14	~	~			
9	0.31	~	0.22	~	~	~	~	~			
10	0.23	0.21	0.26	0.24	0.12	0.20	0.18	0.14			
11	0.30	0.19	0.48	0.70	0.17	0.12	0.19	~			
12	0.29	0.29	0.55	0.58	0.14	0.13	0.07	0.19			
13	0.37	0.17	0.40	0.49	0.04	0.10	0.24	0.12			
14	0.08	0.14	~	0.17	0.05	0.09	0.06	0.11			
15	0.20	0.18	0.38	0.50	0.11	~	~	~			
16	0.59	0.17	0.30	0.63	0.13	0.20	0.22	0.09			
No. of Labs	14	12	11	12	12	11	11	10			
No. of Replicates	1	1	1	1	1	1	1	1			
Average	0.27	0.19	0.32	0.41	0.11	0.13	0.14	0.12			
Std. Deviation	0.13	0.07	0.14	0.19	0.05	0.04	0.06	0.04			

Table F-2. Measured SAM number (unitless) for ASTM E691

~ denotes no data for that entry

APPENDIX G. MEASURED REFERENCE PROPERTIES AFTER STORAGE IN A FOG ROOM (28 DAYS)

Mixture	Average Mass (g)	COV (%)	Average Resistivity (kΩ-cm)	COV (%)	Average f'c (psi)	COV (%)
1A	3968.5	0.6%	17.0	3.1%	7759	2.6%
2A	3957.2	0.7%	12.8	3.3%	6999	2.8%
3A	3870.9	0.6%	15.4	2.6%	6804	4.3%
4A	3872.3	0.6%	14.3	2.0%	6071	2.0%

Table G-1. Measured reference properties after 28 days of storage in a fog room



Figure H-1. Air content versus standard deviation: (a) constant standard deviation and (b) constant coefficient of variation



Figure I-1. SAM number versus standard deviation: (a) constant standard deviation and (b) constant coefficient of variation

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