Precision of Methods for Determining Asphalt Cement Content

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Two data bases provided the statistical information necessary to determine the precision of individual methods of extraction testing with various solvents. Results of this investigation showed no statistical difference between either centrifuge, reflux, or vacuum extraction methods, regardless of the type of solvent used for the test. This precision corroborated the statistics reported in ASTM D2172. Data bases for determining the precision of nuclear asphalt content gauges were created through extensive testing by the University of Nevada-Reno and round robin testing by eight state laboratories. Results from this testing developed a precision statement for ASTM D4125 (nuclear asphalt content gauges). A comparison between the statistics from traditional extraction methods and the nuclear gauges showed that when determining the asphalt content of mixtures with absorptive aggregates (absorption capacity > 2.5 percent) the nuclear gauges appear to be significantly more accurate. Further testing with the nuclear gauges indicated three conclusions. First, the sample temperature did not influence test results when included in the software of the gauges. Second, asphalt content could be determined when mixtures contained latex and polyolefin additives. Third, changes in the gauge environment influenced test results.

The use of end-result specifications implies the ability to measure accurately the quality of the finished product. The qualities of hot-mix asphalt (HMA) pavement stability, durability, and resistance to moisture have historically been tied to the asphalt cement content of the mixture. Flushing, loss of skid resistance, and excessive rutting are signs of too much asphalt cement. Raveling and water damage can indicate that there was too little asphalt in the mix. Assessing the quality of the finished pavement, then, is dependent on how easily and accurately the asphalt cement content of the mixture can be measured.

Several methods for determining asphalt cement content are described by the American Society of Testing and Materials (ASTM). Three methods of extraction are described in ASTM D2172. These are the centrifuge, reflux, and vacuum extractions. One of four specified solvents can be used with any of the three methods. The accuracy of this test method is presented in the form of a precision statement. Although this precision statement presents statistics for testing variation, no distinction is made between any of the three extraction methods or any of the four solvents used with these methods.

Use of the nuclear asphalt cement content gauges is described in ASTM D4125. No precision statement is included in this test method.

To evaluate how accurately asphalt cement content can be measured, research should develop statistical information for each extraction and individual solvents with each extraction method. Statistics should also be developed for the nuclear gauges.

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LITERATURE SEARCH

Test methods for monitoring the asphalt cement content of paving mixtures were developed between 1900 and 1920 (*1*). By 1949, one of the favored methods was the rotarex, a predecessor of today's centrifuge method (*2*). This method requires placing the HMA sample in a centrifuge bowl, pouring room-temperature solvent over it, and spinning the mixture and solvent until the solvent runs clear.

Different versions of reflux extractors were also being evaluated during this period (*3,4*). Reflux extractors heat the solvent; the solvent then evaporates, condenses on the top of the chamber, and drips over the HMA mixture.

Testing on reference samples by multiple laboratories revealed that the reflux method was statistically less variable than the rotarex (*3*). Other reference testing on mixtures with a known asphalt cement content indicated that 0.4 percent of asphalt cement in a mixture could not be recovered (*2*). Approximately 70 percent of laboratories testing the same materials could be expected to report results within 0.5 percent of the actual asphalt cement content (*2*).

The ASTM tentatively accepted procedures for extractions (ASTM D2172) in 1963 (*1*). The standard was officially adopted in 1967. This test method covered the centrifuge, reflux, and vacuum extraction methods.

Several other test methods were proposed in an effort to reduce testing time and test method standard deviations. Asphalt cement content determination by ignition was suggested in 1969 (*5*). The sample is weighed, heated at temperatures high enough to ash the asphalt cement, and then weighed again. The difference in the weights of the sample is the asphalt cement content.

In 1973, a modification of the theoretical maximum specific gravity test (ASTM D2041) was suggested by the Pennsylvania Department of Transportation (*6*). This method uses a weight-volume relationship and the specific gravities of the mixture components to determine the asphalt cement content.

In 1956 the theoretical groundwork was laid for the use of the nuclear asphalt cement content gauges (*7*). By 1969, these gauges had been redesigned and refined sufficiently to produce test results comparable to those of the extraction methods (*8,9*). In 1972, gauges were beginning to have more sophisticated software (*10*). Previously, calculations had to be made by the technician performing the test. These calculations involved determining a mathematical relationship between gauge readings and asphalt cement contents for mixtures with known asphalt cement contents. This relationship was then used to convert subsequent gauge readings into asphalt cement content information. Software advances provided these calculations automatically; thus the chances of calculation errors were reduced.

RESEARCH PROGRAM

The first portion of the research program was designed to evaluate statistical differences in extraction methods (ASTM D2172, Methods A through E) caused by the use of:

1. Centrifuge (Method A), reflux (Methods B, C, D), and vacuum (Method E) extraction methods;
2. Benzene, trichloroethane, trichloroethylene, and methylene chloride solvents;
3. Mixtures prepared with absorptive aggregates.

The data required to evaluate all three extraction methods and the four solvents were obtained from the American Association of State Highway and Transportation Officials (AASHTO) Material Reference Laboratory (AMRL) testing program. AMRL data covered testing performed from 1978 through 1985.

Sets of samples prepared by the University of Nevada-Reno and tested by Reno laboratories (local round robin) were used to evaluate the influence of absorptive aggregates on asphalt cement content determination. Only the centrifuge and reflux extraction methods with trichloroethylene solvent were evaluated. The second portion of the research program was designed to evaluate the nuclear asphalt cement content gauges (ASTM D4125) in order to:

1. Develop a precision statement for the gauges;
2. Compare statistics between the nuclear asphalt cement content gauges and the various extraction methods;
3. Determine the influence of mixture problems on results; and
4. Determine if temperature, additives, or gauge environment influenced the determination of asphalt cement content.

Samples, which were designed to investigate the first three topics, were prepared by the University of Nevada-Reno (UNR) and tested by 10 laboratories from California, Colorado, Illinois, Maryland, Mississippi, Montana, Nevada, and Utah. This program is hereafter referred to as the national round robin.

Samples, which were designed to investigate the influence of sample temperature, additives, and changes in gauge environment, were prepared and tested by the University of Nevada-Reno.

MATERIALS, SAMPLE PREPARATION, AND TESTING

Four testing programs generated the data bases necessary to evaluate the topics just listed. These four programs were the AMRL, local round robin, national round robin, and University of Nevada-Reno. Because the objective of each testing program differed, the materials and/or sample preparation also differed.

AMRL

AMRL test results provided the data for analysis of both the various extraction methods and solvents. AMRL prepared a set of two samples each year to be tested by laboratories participating in its bituminous materials testing program.

Materials

The asphalt grades used to prepare sets of samples varied over the years. Asphalts included in the data base were AC-20, AC-15, AR-4000, and AR-8000. All aggregates used for these various samples had an absorption capacity of less than 1.25 percent.

Sample Preparation

Asphalt grades and aggregate sources differed from year to year. Asphalt cement content and/or aggregate gradations were also varied between the two samples prepared each year; samples were not intended to be replicates. All samples were mixed and prepared by AMRL.

Testing

Laboratories were instructed to perform the tests in accordance with either ASTM D2172 or AASHTO T164. The type of extraction method and solvent used were selected by the individual laboratories. Only one test per sample was performed by participating laboratories.

Local Round Robin

This testing program was designed to evaluate the influence of absorptive aggregates on asphalt cement contents that were determined either by centrifuge or reflux extraction using trichloroethylene.

Materials

Materials used to prepare these samples were

1. An AR-4000 supplied by an Oildale, California, refinery, and
2. A subrounded, partially crushed, river gravel obtained from a quarry in Sparks, Nevada.

This aggregate typically had an absorption capacity greater than 2.5 percent.

Sample Preparation

Individually packaged 2,500-g samples were prepared and distributed to participating local laboratories by the University of Nevada-Reno. An optimum asphalt cement content of 7.0 percent by dry weight of aggregate and a gradation conforming to the Nevada Department of Transportation (NDOT) Type II were used to prepare all mixtures (*11*). All aggregates were sieved into individual fractions and then recombined.

Testing

Of the eight participating laboratories, seven were equipped to perform centrifuge extraction; five laboratories were equipped for reflux extractions. Vacuum extraction was not typically used in this area. Laboratories were instructed to test the samples in accordance with ASTM D2172. They were also asked to use only the trichloroethylene solvent. Moisture content was assumed to be 0; samples were mixed with oven dry aggregate, then immediately sealed after mixing.

National Round Robin

This testing program was designed to evaluate the nuclear asphalt cement content gauges.

Materials

Materials used to prepare these samples were:

1. An AR-4000 supplied by an Oildale, California, refinery;
2. A subrounded, partially crushed, river gravel obtained from a quarry in Sparks, Nevada; and
3. An angular, crushed, low-absorptive (dense) limestone aggregate obtained from an Alabama quarry.

The Sparks, Nevada, aggregate was described in the local round robin section. The Alabama aggregate typically had absorption capacities under 1.0 percent.

Sample Preparation

Because this testing program was designed to evaluate the nuclear asphalt cement content gauge test method (ASTM D4125) as written, it was necessary to provide the laboratories with supplies to prepare calibration point mixtures as well as the UNR premixed test samples of "unknown" asphalt cement content. One set of samples was prepared for each aggregate source.

Each set of samples consisted of

1. 16,000 g of aggregate for preparing the blank and two calibration points;
2. Five quarts of asphalt cement; and
3. Three packages of asphalt-aggregate mixtures (8,560 g each) of undisclosed asphalt cement content (test samples).

The first set of three test samples was prepared with an optimum asphalt cement content of 7.0 percent by dry weight of aggregate. The aggregate was a rounded, partially crushed, absorptive aggregate. The data base created by testing these samples was used to develop a suggested precision statement for ASTM D4125.

The second set of test samples was prepared with an excess asphalt cement content of 7.0 percent by dry weight of aggregate; the angular, dense aggregate; and the same NDOT gradation as was used to generate a mixture exhibiting two noticeable problems. First, this mixture only partially filled the gauges' sample pan at the target weight. Second, the asphalt cement tended to pool in the bottom of the pan. This pooling created an uneven distribution of asphalt throughout the test sample. The data base created by these samples was used to evaluate the influence of mixture problems on asphalt cement content determination.

Testing

Participating laboratories were asked to prepare two calibration samples for each aggregate source and to analyze appropriate test samples as directed in ASTM D4125. Asphalt cement contents for the calibration points were 6 and 8 percent by dry weight of aggregate.

University of Nevada-Reno

This testing program was designed to evaluate the influence of sample temperature, gauge environment, and asphalt cement additives on the nuclear asphalt cement content gauges.

Materials

The same samples prepared for the national round robin were used for evaluating the influence of sample temperature and changes in gauge environment.

The AR-4000 previously mentioned was used to assess the effect of additives in mixtures. The AR-4000 was modified with 5 percent styrene-butadine rubber latex and 3 percent polyolefin plastic (percent by weight of asphalt). Only the rounded river gravel was used; the gradation was the same as for the national round robin.

Sample Preparation

No new samples were prepared for monitoring the influence of sample temperature and changes in gauge environment. The samples prepared for the national round robin were used after testing for that phase was completed.

A new set of calibration points and three test samples were prepared with the modified asphalt cement. Seven percent of modified asphalt cement (dry weight of aggregate) was used with the rounded, absorptive aggregate to prepare these test samples. Calibration points were mixed with the modified asphalt cement at 6 and 8 percent (dry weight of aggregate).

Testing

Sample temperature effects were evaluated using both sets of samples prepared by the University of Nevada-Reno for the national round robin. Sample temperatures were monitored as they cooled and were periodically retested for asphalt cement content. Two gauges were used for testing:

1. Troxler 3241-B and
2. CPN AC-2.

Changes in gauge environment were evaluated by placing one of the samples from the national round robin, cooled to room temperature, in each of the two gauges just listed. Loose HMA samples of approximately 8,000 g stored in plastic concrete cylinders were then placed next to each gauge, and the asphalt cement content of sample already in the gauge was redetermined. The number of loose mix samples next to the gauges ranged from one to five.

Samples prepared with the modified asphalt cement were tested in accordance with the ASTM D4125 test method.

STATISTICS

Statistics for test results are commonly expressed as within- and between-laboratory standard deviations. These standard deviations are also referred to as repeatability and reproducibility, respectively.

Determining Within- and Between-Laboratory Statistics

The method used to calculate these statistics is dependent on how the data base was developed. Data bases for this research project were developed in one of two ways. Laboratories tested two or more samples; either none of the samples were replicates or two or more of them were replicates.

When data bases are constructed with no replicates, within-laboratory standard deviations are calculated using the differences between two test results generated at the same time by the same laboratory. The between-laboratory standard deviation is calculated for each material; each laboratory contributes only one data point. These statistics are used for analysis of the AMRL and the University of Nevada-Reno data bases.

For data bases with one or more replicates, calculation of within- and between-laboratory standard deviations is described by ASTM C670 and ASTM C802 (*12*). Within-laboratory standard deviation in this case is based on the average variance between replicates tested by each laboratory. Between-laboratory standard deviation is a function of variance between each set of replicates and the within-laboratory variance per sample. These statistics were used for analysis of the local and national round robin data bases.

Determining If Statistics from Two Different Populations Are Similar

Once the appropriate within- and between-laboratory standard deviations have been calculated for each variable (e.g., each method of extraction) within each data base, statistical comparisons can be made. Data bases with different variables are compared by calculating an *F*-value. The *F*-value is a ratio of variances (i.e., standard deviation squared) and is calculated by

$$F\text{-value (calculated)} = s21 / s22$$

where

$s21$ equals the largest of two variances being evaluated and $s22$ equals the variance of the other population.

Once the *F*-value has been calculated, a table *F*-value is found from a typical table. Variables needed to use these tables are

1. n, the population size;
2. v, the degrees of freedom;
3. Level of confidence; and
4. Level of significance.

The population size, n, is the number of samples tested. The degrees of freedom, v, is n minus 1. The degrees of freedom are used to enter the statistical tables (*13*).

The level of confidence and significance are related. A level of confidence is chosen by the investigator and typically is either 95 or 99 percent. This is a measure of how sure the investigator is that the final conclusion is correct. The level of significance is a measure of risk. If the investigator is 95 percent confident the conclusion is correct, he or she is also willing to risk a 5 percent chance of a wrong conclusion. This 5 percent is the level of significance.

A conclusion is drawn by comparing the two *F*-values. If the calculated value is greater than the table value, the variances are different (*13*). That is, the differences between the two populations change the variance. If the calculated value is less than the table value, the variances are the same for both populations. That is, the differences between the two populations do not appear to affect the variances.

EVALUATION OF TEST RESULTS

The data bases generated by the four testing programs outlined in the previous section were sorted and analyzed in order to evaluate

1. Differences between extraction methods;
2. Differences between selected solvents used with various extraction methods;
3. Influence of absorptive aggregates on test results for selected extraction methods;
4. Precision statement for the nuclear asphalt cement content gauges (ASTM D4125);
5. Comparison of the nuclear asphalt cement content gauge and extraction methods; and
6. Influence of sample temperature, gauge environment, and additives on nuclear asphalt cement content readings.

Differences Between Extraction Methods

Each yearly set of two AMRL samples was separated by methods of extraction. These yearly variances for each test

TABLE 1 STATISTICS FOR CENTRIFUGE, REFLUX, AND VACUUM EXTRACTIONS

Extraction Method	Type of Statistic	Variance	Standard Deviation
Centrifuge	Within-Laboratory	0.0467	0.216
	Between-Laboratory	0.0472	0.217
Reflux	Within-Laboratory	0.0342	0.185
	Between-Laboratory	0.0515	0.227
Vacuum	Within-Laboratory	0.0426	0.206
	Between-Laboratory	0.0489	0.221

TABLE 2 COMPARISON OF CENTRIFUGE, REFLUX, AND VACUUM EXTRACTIONS

Extraction Method	Type of Statistic	Degrees of Freedom	Calc. F-Value	Table F-Value	Conclusion
Centrifuge vs. Reflux	Within-Laboratory	19 19	1.366	2.17	No Diff.
	Between-Laboratory	37 39	1.091	1.74	No Diff.
Centrifuge vs. Vacuum	Within-Laboratory	19 20	1.096	2.14	No Diff.
	Between-Laboratory	41 39	1.036	1.72	No Diff.
Reflux vs. Vacuum	Within-Laboratory	20 19	1.246	2.16	No Diff.
	Between-Laboratory	37 41	1.053	1.73	No Diff.

method were then averaged (Table 1). Because the number of samples varied from year to year, a weighted average was used to average the variances.

The average of the within- and between-laboratory standard deviations shown in Table 1 are 0.20 and 0.22, respectively. ASTM D2172 reports these same standard deviations, for any method, as 0.18 and 0.29, respectively. The within-laboratory standard deviations are almost identical. The between-laboratory values suggest that a slight, but statistically significant, difference may exist between the AMRL and ASTM statistics.

Table 2 presents the calculated and table F-values for comparisons between the three extraction methods. The conclusion is that there is no statistical difference between the three extraction methods.

Differences Between Assorted Solvents with Various Extraction Methods

The AMRL data bases used in the previous section were sorted further by solvent and extraction method. The variances for these variables are shown in Table 3.

Table 4 presents the calculated and table F-values. There is no difference between the various solvents used with any of the extraction methods.

Influence of Absorptive Aggregates on Test Results for Selected Extraction Methods

The variance and standard deviations for both centrifuge and reflux extraction are shown in Table 5. A comparison of these

TABLE 3 STATISTICS FOR ASSORTED SOLVENTS WITH VARIOUS EXTRACTION METHODS

Extraction Method	Solvent	Type of Statistic	Variance	Standard Deviation
Centrifuge	Benzene	Within-Lab.	0.0088	0.0938
		Between-Lab.	0.0154	0.1241
	Trichloroethane	Within-Lab.	0.0520	0.2280
		Between-Lab.	0.0461	0.2147
	Trichloroethylene	Within-Lab.	0.0499	0.2234
		Between-Lab.	0.0553	0.2352
Reflux	Benzene	Within-Lab.	0.0499	0.2234
		Between-Lab.	0.0349	0.1868
	Trichloroethane	Within-Lab.	0.0388	0.1970
		Between-Lab.	0.0572	0.2392
	Trichloroethylene	Within-Lab.	0.0305	0.1747
		Between-Lab.	0.0501	0.2238
Vacuum	Methelene Chloride	Within-Lab.	0.0434	0.2083
		Between-Lab.	0.0481	0.2193
	Trichloroethane	Within-Lab.	0.0294	0.1715
		Between-Lab.	0.0511	0.2261
	Trichloroethylene	Within-Lab.	0.0500	0.2236
		Between-Lab.	0.0482	0.2200

standard deviations with those obtained from the AMRL data base is also shown in Table 6. The absorptive aggregate creates statistically significant changes in the between-laboratory standard deviations for both the reflux and centrifuge extraction methods.

It is suggested that the precision statement for ASTM D2172 be expanded to reflect these additional statistics. The suggested form for the addition is:

Test and Type Index	*Standard Deviation (1S)*	*Acceptable Range of Two Results (D2S)*
Tests with Mixtures with Porous Aggregate Single-operator precision Method A (Centrifuge)	0.55	1.56
Method B, C, or D (Reflux)	0.20	0.57
Multilaboratory precision Method A (Centrifuge)	0.55	1.56
Method B, C, or D (Reflux)	0.31	0.88

Precision of the Nuclear Asphalt Cement Content Gauges

A close examination of the raw data indicated that three of the laboratories mixed the calibration points at 6 and 8 percent asphalt by total weight of mix instead of by dry weight of aggregate. The reported test results were corrected to reflect the asphalt cement content by dry weight of aggregate. The two sets of statistics generated by these corrected data are presented in Table 7. The first set of statistics represents mixtures prepared at an optimum asphalt cement content. The second set of statistics represents mixtures with problems, such as partially full sample pans at the target weight and pooling asphalt.

The ratios of within- and between-laboratory standard deviations (calculated F-values) for these two mixtures are 2.525 and 1.178, respectively. These calculated F-values, compared with a table F-value of 1.85, show that the problem mix significantly decreases the within-laboratory standard deviations. There is no significant between-laboratory difference. Problems with the mixture do not adversely influence the statistics. It should be kept in mind that the calibration points also reflected these problems.

The suggested form for a precision statement for ASTM D4125, based on the statistics for the optimum asphalt cement content mixture, is:

Test and Type Index	*Standard Deviation (1S)*	*Acceptable Range of Two Results (D2S)*
Single-operator precision	0.16	0.45
Multilaboratory precision	0.23	0.65

Comparison of the Extraction Methods (ASTM D2172) and the Nuclear Asphalt Cement Content Gauges (ASTM D4125)

The within- and between-laboratory standard deviations presented in the ASTM D2172 precision statement for the extrac-

TABLE 4 COMPARISON OF SELECTED SOLVENTS WITH VARIOUS EXTRACTION METHODS

Comparison	Type of Statistic	Degrees of Freedom	Calc. F-Value	Table F-Value	Conclusion
Centrifuge:					
Benzene vs. Trichloroethane	Within-Lab.	8,1	5.909	239	No Diff.
	Between-Lab.	17,3	2.994	8.68	No Diff.
Benzene vs. Trichloroethylene	Within-Lab.	8,1	5.671	239	No Diff.
	Between-Lab.	17,3	3.591	8.68	No Diff.
Trichloroethane vs. Trichloroethylene	Within-Lab.	8,8	1.042	3.44	No Diff.
	Between-Lab.	17,17	1.200	2.28	No Diff.
Reflux:					
Benzene vs. Trichloroethane	Within-Lab.	1,7	1.286	5.59	No Diff.
	Between-Lab.	15,3	1.639	8.70	No Diff.
Benzene vs. Trichloroethylene	Within-Lab.	1,8	1.636	5.32	No Diff.
	Between-Lab.	17,3	1.436	8.68	No Diff.
Trichloroethane vs. Trichloroethylene	Within-Lab.	7,8	1.272	3.50	No Diff.
	Between-Lab.	15,17	1.142	2.31	No Diff.
Vacuum:					
Methelene Chloride vs. Trichloroethane	Within-Lab.	7,4	1.476	6.09	No Diff.
	Between-Lab.	9,15	1.062	2.59	No Diff.
Methelene Chloride vs. Trichloroethylene	Within-Lab.	7,7	1.152	3.79	No Diff.
	Between-Lab.	11,9	1.002	3.10	No Diff.
Trichloroethane vs. Trichloroethylene	Within-Lab.	7,4	1.701	6.09	No Diff.
	Between-Lab.	9,11	1.060	2.90	No Diff.

TABLE 5 STATISTICS FOR EXTRACTION METHODS WITH ABSORPTIVE AGGREGATES, LOCAL ROUND ROBIN

Extraction Method	Type of Statistic	Variance	Standard Deviation
Centrifuge	Within-Lab.	0.30	0.55
	Between-Lab.	0.31	0.55
Reflux	Within-Lab.	0.04	0.20
	Between-Lab.	0.10	0.31

tion methods are 0.18 and 0.29, respectively. The same standard deviations for the AMRL data were 0.20 and 0.22, respectively. The nuclear asphalt cement content gauges produce within- and between-laboratory standard deviations of 0.16 and 0.23, respectively (Table 7). The nuclear gauges appear to reduce the within-laboratory standard deviations. The between-laboratory standard deviation for these gauges is virtually identical to that for extractions (AMRL).

Because the optimum asphalt cement content mixtures were prepared with the same rounded, absorptive aggregates used for the local round robin, these statistics can also be compared. Recall that mixtures prepared with this aggregate significantly increased the between-laboratory standard deviations for both the centrifuge and reflux extraction methods. The between-laboratory standard deviations were 0.55 for centrifuge and 0.31 for reflux extraction (Table 5). The same mixture used in the nuclear asphalt cement content gauges produced a between-laboratory standard deviation of 0.23. A substantial improvement in between-laboratory standard deviations is gained when the nuclear gauge is used in place of either the centrifuge or reflux extraction methods for mixtures with this type of aggregate.

TABLE 6 COMPARISON OF VARIOUS EXTRACTION METHODS WITH MIXTURES, BOTH ABSORPTIVE AND LOW-ABSORPTIVE AGGREGATES

Comparison	Type of Statistic	Degrees of Freedom	Calc. F-Value	Table F-Value	Conclusion
Absorptive Agg. Centrifuge vs. Reflux (Local Round Robin)	Within-Lab.	30 30	7.50	1.87	Difference Between Extraction Methods
	Between-Lab.	30 30	3.10	1.87	Difference Between Extraction Methods
Centrifuge Extraction Dense vs. Absorpt. Agg.*	Within-Lab.	29 19	6.42	2.08	Agg. cause changes in statistics
	Between-Lab.	29 37	6.57	1.78	Agg. cause changes in statistics
Reflux Extraction Dense vs. Absorpt. Agg.*	Within-Lab.	29 19	1.17	2.08	No diff. in statistics
	Between-Lab.	29 39	1.94	1.73	Agg. cause changes in statistics

* The AMRL statistics for the appropriate extraction method and trichloroethylene solvent were used to represent statistics for mixtures with dense aggregates.

TABLE 7 STATISTICS FOR NUCLEAR ASPHALT CEMENT CONTENT GAUGES, NATIONAL ROUND ROBIN

Material Property	Type of Statistic	Variance	Standard Deviation
Rounded, Absorptive Aggregate with Optimum Asphalt Content	Within-Lab. Between-Lab.	0.0250 0.0518	0.1582 0.2277
Angular, Dense Aggregate with Excess Asphalt Content	Within-Lab. Between-Lab.	0.0099 0.0610	0.0993 0.2470

Influence of Temperature, Gauge Environment, and Asphalt Cement Modifiers

Testing for these evaluations was performed by the University of Nevada-Reno. Two gauges were used:

1. Troxler 3241-B and
2. CPN AC-2.

The repeatability of each of these gauges was established prior to any testing. The test results for each gauge are shown in Table 8. The Troxler and the CPN gauges had standard deviations from 0.01 to 0.02, and from 0.03 to 0.04 percent asphalt, respectively.

Temperature

Tables 9 through 11 show samples tested at various temperatures. The test results in Tables 9 and 10 were generated by the University of Nevada-Reno. The test results in Table 11 were provided by Laboratory 2 from the national round robin.

TABLE 8 REPEATABILITY FOR BOTH GAUGES USED BY THE UNIVERSITY OF NEVADA–RENO

Gauge	Duration of Reading (Minutes)	Asphalt Cement Content Readings, %	Standard Deviation (% AC)
Troxler 3241-B*	1	6.80, 6.81, 6.82, 6.84, 6.78	0.02
	4	6.83, 6.81, 6.82, 6.78, 6.81	0.02
	16	6.81, 6.83, 6.82, 6.84, 6.81	0.01
CPN AC-2**	1	6.11, 6.18, 6.17, 6.17, 6.12	0.03
	4	6.11, 6.11, 6.22, 6.18, 6.21	0.04
	16	6.12, 6.16, 6.16, 6.16, 6.21	0.03

* Angular, Dense Aggregate, 7.0% AC
** Angular, Dense Aggregate, 6.0% AC (Calibration Pan)

TABLE 9 VARIATIONS IN ASPHALT CEMENT CONTENT READING WITH CHANGES IN SAMPLE TEMPERATURE; UNIVERSITY OF NEVADA–RENO DATA MIXTURES WITH ROUNDED, ABSORPTIVE AGGREGATE, OPTIMUM ASPHALT CEMENT CONTENT, 7.0 PERCENT

Troxler 3241-B		CPN AC-2	
Temperature (F)	Asphalt (%)	Temperature (F)	Asphalt (%)
Sample 1-Four Min. Reading		Sample 1-Four Min. Reading	
225	6.86	100	7.16
100	6.89	85	7.16
85	6.95		
Sample 1-One Min. Reading		Sample 1-One Min. Reading	
225	6.84	100	7.12
100	6.90	85	7.21
Sample 2-Four Min. Reading		Sample 2-Four Min. Reading	
140	6.80	190	6.84
100	6.91	90	6.98
80	6.94		
Sample 2-One Min. Reading		Sample 2-One Min. Reading	
140	6.83	190	6.84
100	6.84	90	6.98
80	6.89		
Sample 3-Four Min. Reading		Sample 3-Four Min. Reading	
180	6.83	200	6.93
115	6.79	130	7.06
		100	7.04
Sample 3-One Min. Reading		Sample 3-One Min. Reading	
180	6.83	200	7.01
115	6.81	130	7.12
		100	7.00

TABLE 10 VARIATIONS IN ASPHALT CEMENT CONTENT READING WITH CHANGES IN SAMPLE TEMPERATURE; UNIVERSITY OF NEVADA-RENO DATA MIXTURES WITH ANGULAR, DENSE AGGREGATE, EXCESS ASPHALT CEMENT (7.0 PERCENT)

Troxler 3241-B		CPN AC-2	
Temperature (F)	Asphalt (%)	Temperature (F)	Asphalt (%)
Sample 1		Sample 1	
Not Available		200	7.12
		100	7.18
		90	7.17
Sample 2		Sample 2	
Not Available		195	7.05
		120	7.09
		100	7.11
Sample 3		Sample 3	
Not Available		185	6.96
		140	7.02
		110	7.13

TABLE 11 VARIATIONS IN ASPHALT CONTENT READING WITH CHANGES IN SAMPLE TEMPERATURE; DATA SUPPLIED BY LABORATORY 2

Rounded, Absorptive Agg. Optimum Asphalt Content, 7.0%		Angular, Dense Agg. Excess Asphalt Content, 7.0%	
Temperature (F)	Asphalt (%)	Temperature (F)	Asphalt (%)
Sample 1		Sample 1	
220	6.87	190	7.02
68	6.85	70	7.05
Sample 2		Sample 2	
250	6.67	135	6.90
68	6.57	70	6.90
Sample 3		Sample 3	
215	6.50	215	6.89
68	6.51	70	6.88

None of the test results indicates any trends in asphalt cement content readings versus sample temperature. It should be noted that the gauges used to generate these results were equipped with software that required the operator to enter the sample temperature.

Changes in Gauge Environment

Table 12 shows the results of operating the gauges with various quantities of HMA materials stacked near the gauges. Gauges were first calibrated; then asphalt concrete materials, stored in plastic concrete cylinders, were placed near the gauges. There was approximately 8,000 g of material in each canister. Small but discernible changes occur as progressively more material was placed near the gauges. Placing five canisters (approximately 40,000 g of material) near the gauges increases the asphalt cement content readings from 6.96 to 7.07 on the Troxler gauge and from 6.99 to 7.12 on the CPN gauge.

Modified Asphalt Cements

Table 13 presents the results of testing mixtures with modified asphalts. The same set of calibration pans and test samples were used in both gauges. Test results indicate a tendency for the Troxler gauge to read a slightly lower asphalt cement content than the CPN. This difference was not noticeable when identical unmodified samples were tested for gauge environment study (Table 12). Although this trend appears to be consistent between these sets of test results, the variation is well within the acceptable range of two test results (single-operator) variation of 0.45 percent presented in the suggested precision statement.

TABLE 12 VARIATIONS IN ASPHALT CEMENT CONTENT READING WITH CHANGES IN GAUGE ENVIRONMENT; ONE SAMPLE USED IN BOTH GAUGES: ROUNDED, ABSORPTIVE AGGREGATE, OPTIMUM ASPHALT CEMENT CONTENT, 7.0 PERCENT

Number of Samples Near Gauge	Duration of Reading (Minutes)	Asphalt Cement Content, %	
		Troxler 3241-B	CPN AC-2
None	1	6.92	6.98
	4	6.95	6.99
	16	7.00	7.00
One Sample	1	7.02	6.99
	4	7.01	6.99
	16	7.01	7.01
Two Samples	1	7.04	7.01
	4	7.03	7.06
	16	7.04	7.01
Five Samples	1	7.09	7.11
	4	7.08	7.12
	16	7.05	7.12

TABLE 13 INFLUENCE OF ASPHALT CEMENT ADDITIVES ON ASPHALT CEMENT CONTENT READINGS; SAMPLES PREPARED WITH 5 PERCENT LATEX AND 3 PERCENT PLASTIC (WEIGHT OF ASPHALT CEMENT) AND ROUNDED, ABSORPTIVE AGGREGATE

Sample Number	Duration of Reading (Minutes)	Asphalt Cement Content, %	
		Troxler 3241-B	CPN AC-2
Sample 1	1	6.93	6.87
	4	6.88	7.02
	16	6.83	6.97
Sample 2	1	6.84	7.22
	4	6.88	7.09
	16	6.83	7.02
Sample 3	1	6.98	6.99
	4	6.88	7.05
	16	6.83	7.04

The use of modified asphalts in mixtures appears to have little influence on the reliability of the gauge readings when the calibration points are prepared with the same asphalt.

CONCLUSION

The conclusions of this research program follow.

1. There is no statistical difference between the results of centrifuge, reflux, and vacuum extraction methods;
2. There is no statistical difference between the results from any particular solvents used in this study with any of the three extraction methods;
3. The existing ASTM D2172 precision statement is valid for any extraction method with any of the solvents for mixtures with low-absorptive aggregates;
4. The existing ASTM D2172 precision statement needs to be expanded to include statistics for mixtures with absorptive aggregates;
5. The within- and between-laboratory standard deviations for the nuclear asphalt cement content gauge (ASTM D4125) are 0.16 and 0.23, respectively;
6. Problems with mixtures do not affect the accuracy of the nuclear asphalt cement content gauges when these same problems are also present in the calibration samples;
7. Changes in gauge environments can change the asphalt cement content reading; and

8. Some modified asphalt cements do not appear to influence the accuracy of the nuclear asphalt cement content gauges when the calibration points are also prepared with the modified asphalt.

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