

Improved Rice Method for Determining Theoretical Maximum Specific Gravity of Asphalt Paving Mixtures

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The Rice method (ASTM D2041) is used for determining the theoretical maximum specific gravity of asphalt paving mixtures, which is one of the main test parameters used for mix design and construction quality control. The repeatability and reproducibility of the Rice method is not considered satisfactory, especially when highly absorptive aggregates are used in the mixture. Such aggregates can absorb water during the vacuuming phase of the Rice method. This necessitates the use of a supplementary (dry-back) procedure that is even more prone to testing errors. There is a need to improve the Rice method to avoid these problems. Two dense-graded asphalt paving mixtures (representing low- and high-absorption aggregates) and one AC-20 asphalt cement were used. Three factors (temperature, residual pressure, and vacuuming time) affecting the results from the Rice method were investigated at three levels each, involving some 108 tests. From these test data, optimum levels have been recommended for temperature, residual pressure, and vacuuming time. Use of these optimum levels is expected to improve the reproducibility of the Rice method, especially when highly absorptive aggregates are used in the asphalt paving mixtures. The improved Rice method is also likely to minimize the necessity of using the supplementary procedure.

Theoretical maximum specific gravity of hot mix asphalt (HMA) mixtures is used in all stages of HMA design, construction, and evaluation. The procedure for determining the theoretical maximum specific gravity of HMA mixtures was originally developed by Rice (1,2); it is standardized as ASTM D2041 (Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixtures). It is one of the main test parameters used for mix design and construction quality control. Besides the void parameters of an HMA mixture, the amount of asphalt cement absorbed by the aggregate is also computed by the use of theoretical maximum specific gravity as determined by ASTM D2041.

Although this is one of the most critical test parameters in the design of HMA mixtures, its repeatability and reproducibility have generally been unacceptable. In July 1983, the task force on revision of ASTM D2041 (the Rice specific gravity method) recommended that the water be made to boil (through suitable combinations of saturation temperature and pressure) in the vessel when subjecting the specimen to vacuum for removal of air. However, no test data were reported to justify the recommendation, and it was not made a part of the test method.

The situation is further complicated when highly absorptive aggregates are used in HMA mixtures and there is the po-

tential of water's being absorbed by the aggregate during the vacuuming phase. The current ASTM method specifies a minimum vacuum level [maximum residual pressure of 30 mm of mercury (Hg) or less]. Therefore, one can use a high vacuum level and increase the potential for water absorption. The current ASTM test method suggests the use of a supplemental procedure to correct for this situation. However, this procedure is time-consuming, and its repeatability and reproducibility have not been established.

Alternative techniques for determining the theoretical maximum specific gravity have also been proposed and are in use. The U.S. Army Corps of Engineers developed and has used the bulk impregnated specific gravity in the design and control of asphalt paving mixtures (3,4). One limitation of this method, however, is the possible difficulty of removing air bubbles entrapped in the asphalt cement when both coarse and fine aggregates are added to molten asphalt.

Franco and Lee have recently evaluated the viability of using an air meter for determining the maximum specific gravity of HMA mixtures (5). The air meter has normally been used for determining the percent of air entrained in portland cement concrete. The so-called pressure method works on the principle of Boyle's law. A weighed sample of HMA mix is introduced into the bowl of the air meter (Type B air meter as specified in AASHTO T152) and water is introduced to fill the meter to the capacity (no attempt is made to remove the entrapped air). The filled air meter is weighed and the weight of water obtained. The air content of the meter is then determined in accordance with AASHTO T152. Back calculations are then performed, and the volume of the HMA mix is determined. Further work is needed to improve the design of the air meter so that its consistency and sensitivity are acceptable.

This study was aimed at refining the current Rice method (ASTM D2041). Two aggregates and one asphalt cement were used. Three factors—namely, temperature, residual pressure, and vacuuming time—were investigated to determine their optimum levels. The materials used in the study, the experimental plan, test results, and conclusions are presented in the following.

MATERIALS USED

Aggregates

All the materials used in this study were obtained from the Strategic Highway Research Program's (SHRP) Materials

Reference Library (MRL). The following two different aggregates were used:

- RD—Frederick limestone (bulk specific gravity = 2.713, water absorption = 0.38 percent)
- RB—Watsonville granite (bulk specific gravity = 2.692, water absorption = 1.68 percent)

Aggregate RD was used in the first phase of the study in which three factors—temperature, residual pressure, and vacuuming time—were investigated for their optimum levels. This aggregate was selected because it had the lowest water absorption of all the SHRP MRL aggregates and its use would reduce the variation of test results due to water absorption during the vacuuming operation. Aggregate RB, which was used in Phase 2 of this study, was selected to represent a high-absorption aggregate. Initially, RC was tried, which is the highest-absorption SHRP MRL aggregate. However, during vacuuming at low residual pressures (high vacuum) while running the Rice method, the water in the flask became muddy because of suction of fines from the coated aggregate particles. As a result, the supplemental procedure was in error. Using a high-absorption aggregate such as RB was necessary for amplifying the difference in the values of the theoretical maximum specific gravity before and after running the supplemental procedure. The washed gradations of the mixes used are given in Table 1.

TABLE 1 Washed Gradations of Mixes Used

Sieve Size	Percent Passing for Aggregate	
	RB	RD
1/2 in	100.0	100.0
3/8 in	81.1	95.8
No. 4	54.1	50.3
No. 8	48.6	40.4
No. 16	35.0	28.7
No. 30	26.8	20.0
No. 50	18.5	14.7
No. 100	11.0	11.5
No. 200	6.1	9.5

Asphalt Cements

Only one asphalt cement (AAM-1, West Texas) was used in this study, and it was an AC-20. It was selected because AC-20 is the most widely used viscosity-graded asphalt cement in the United States.

TESTING PLAN

This study was divided in two phases. Phase 1 involved the use of RD, the SHRP MRL aggregate with the least water absorption, in order to avoid the supplemental procedure of the Rice method. The temperature, residual pressure, and vacuuming time were all investigated at three levels each. The levels of the factors tested were

- Temperature—69, 77, and 85°F;
- Residual pressure—16, 23, and 30 mm Hg; and
- Vacuuming time—5, 10, and 15 min.

The respective combinations of temperature and residual pressure—namely, 69°F and 16 mm Hg, 77°F and 23 mm Hg, and 85°F and 30 mm Hg—were selected to ensure that the water boiled continuously during the vacuuming phase of the Rice procedure. Three replicates were run at each treatment combination. The design was a 3³ design with three replicates, giving a total of 81 tests. The replicates were considered as blocks.

For Phase 2, the factor temperature was dropped on the basis of the results from Phase 1. The other two factors, that is, residual pressure and vacuuming time, were included at three levels each. The levels used for these factors were the same as for Phase 1. Three replicates were run for each treatment combination and were treated as blocks. The design was a 3² design with three replicates, giving a total of 27 tests.

DISCUSSION OF RESULTS

The results for Phase 1 of the study are reported in Table 2. An analysis of variance (ANOVA) was carried out; it is re-

TABLE 2 Rice Specific Gravities Using Aggregate RD

Temperature, °F	Res. Pressure, mm Hg	69			77			85		
		16	23	30	16	23	30	16	23	30
V a c u u m	5	2.501	2.495	2.503	2.492	2.495	2.499	2.495	2.495	2.500
		2.493	2.498	2.501	2.491	2.495	2.492	2.491	2.494	2.493
		2.492	2.493	2.500	2.491	2.490	2.492	2.493	2.495	2.494
T i m e	10	2.495	2.495	2.501	2.491	2.493	2.494	2.493	2.495	2.496
		2.495	2.496	2.496	2.496	2.491	2.493	2.495	2.495	2.495
		2.496	2.498	2.496	2.493	2.490	2.496	2.497	2.494	2.501
m i n	15	2.497	2.499	2.499	2.499	2.493	2.491	2.494	2.493	2.502
		2.496	2.498	2.497	2.496	2.491	2.493	2.495	2.494	2.499
		2.496	2.501	2.500	2.502	2.499	2.502	2.497	2.500	2.499
		2.496	2.499	2.500	2.497	2.495	2.500	2.497	2.497	2.496
		2.497	2.498	2.497	2.498	2.496	2.495	2.495	2.497	2.494
		2.496	2.499	2.499	2.499	2.497	2.499	2.496	2.498	2.496

ported in Table 3. Temperature (*A*), residual pressure (*B*), and vacuuming time (*C*) were all found to be significant at $\alpha = 0.05$. Only one interaction, $A \times C$ (temperature \times vacuuming time), was found to be significant at $\alpha = 0.05$. By looking at the averages reported in Table 2 for each factor, it can be seen that average theoretical maximum specific gravity does not vary much at the various levels of all factors. The error variance is found to be 0.00001, giving a standard deviation of 0.00228. This gives a single-operator precision for the experiment of 0.007, which is less than the current ASTM value of 0.011. Moreover, the range of the values for the entire experiment is $2.490 - 2.502 = 0.012$, which is about the same as the current ASTM single-operator precision.

For Phase 1 of the study, the objective was to maximize the value of the theoretical maximum specific gravity. The optimal conditions for all the factors are selected as follows.

Without Considering Interactions

The cumulative averages (average of 27 observations) for all the factors are shown in Table 4.

Observing the cumulative average values of the theoretical maximum specific gravity, the optimal levels of factors, based on the highest value, are selected as

- Temperature—69°F,
- Residual pressure—30 mm Hg, and
- Vacuuming time—15 min.

Considering Interaction $A \times C$

The cell averages for interaction $A \times C$ are as follows:

Temperature (°F)	Vacuuming Time (min)		
	5	10	15
69	2.497	2.497	2.498
77	2.493	2.494	2.498
85	2.494	2.496	2.497

Observing the cell averages, the optimal level for Factor *C* (vacuuming time) is confirmed to be 15 min. However, for Factor *A* (temperature), two levels are indicated: 69 and 77°F. Because there is not much variation in results, and moreover, because 77°F is a representative indoor temperature and is commonly used for most of the indoor testing, it was selected as the optimal temperature.

TABLE 3 ANOVA for Rice Gravities Using Aggregate RD

Source	df	SS	MS	F _o	F _{Crit.}
Total	80	0.00081	-	-	-
Temperature (A)	2	0.00009	0.00005	8.72	3.18 *
Res. Pressure (B)	2	0.00006	0.00003	5.42	3.18 *
Vacuum Time (C)	2	0.00012	0.00006	11.69	3.18 *
AxB	4	0.00003	0.00001	1.53	2.56
AxC	4	0.00006	0.00002	3.08	2.56 *
BxC	4	0.00004	0.00001	2.03	2.56
AxBxC	8	0.00008	0.00001	1.99	2.13
Error	52	0.00027	0.00001	-	-

* Significant at $\alpha = 0.05$.

TABLE 4 Cumulative Averages for All Factors

Level	Cumulative Average
Temperature (°F)	
69	2.497
77	2.495
85	2.496
Residual pressure (mm Hg)	
16	2.495
23	2.496
30	2.497
Vacuuming time (min)	
5	2.495
10	2.496
15	2.498

The optimal levels for the factors selected on the basis of Phase 1 of the study, therefore, are

- Temperature—77°F,
- Residual pressure—30 mm Hg, and
- Vacuuming time—15 min.

As a consequence of the results from Phase 1, it was decided to drop the factor temperature from the design for Phase 2 of the study. For Phase 2, the high-water-absorption aggregate RB was used.

The results from Phase 2 experiments are reported in Table 5. The reported results include the values for theoretical maximum specific gravity both before and after running the supplementary procedure. The ANOVAs for this phase are reported in Tables 6 and 7, respectively, for difference in theoretical maximum specific gravity values before and after running the supplementary procedure and theoretical maximum specific gravity values before running the supplementary procedure.

Differences

Here the Factor *A* (residual pressure) and the interaction $A \times B$ (residual pressure \times vacuuming time) are found to be significant at $\alpha = 0.05$. The objective in this case was to minimize the differences. To determine the optimal levels, the cumulative averages for factor *A* are reported as

Level (mm Hg)	Cumulative Average
16	0.008
23	0.007
30	0.004

The optimal levels of factors, without considering interactions, are selected as

- Residual pressure—30 mm Hg, and
- Vacuuming time—Any level.

Now, because the interaction $A \times B$ was also significant, we should take a look at the cell averages:

Residual Pressure (mm Hg)	Vacuuming Time (min)		
	5	10	15
16	0.007	0.010	0.008
23	0.010	0.003	0.009
30	0.003	0.006	0.003

TABLE 5 Rice Specific Gravities Using Aggregate RB

Residual Pressure (A), mm Hg	Vacuuming Time (B), min.								
	5			10			15		
	Before	After	Difference	Before	After	Difference	Before	After	Difference
16	2.529	2.524	0.005	2.533	2.523	0.010	2.534	2.528	0.006
	2.527	2.520	0.007	2.524	2.514	0.010	2.531	2.524	0.007
	2.529	2.519	0.010	2.524	2.515	0.009	2.532	2.521	0.011
Avg.	2.528	2.521	0.007	2.527	2.517	0.010	2.532	2.524	0.008
23	2.518	2.508	0.010	2.532	2.529	0.003	2.530	2.521	0.009
	2.520	2.510	0.010	2.526	2.524	0.002	2.531	2.523	0.008
	2.523	2.513	0.010	2.526	2.522	0.004	2.536	2.525	0.011
Avg.	2.520	2.510	0.010	2.528	2.525	0.003	2.532	2.523	0.009
30	2.529	2.527	0.002	2.524	2.519	0.005	2.532	2.530	0.002
	2.522	2.518	0.004	2.527	2.519	0.008	2.528	2.526	0.002
	2.524	2.521	0.003	2.520	2.516	0.004	2.527	2.523	0.004
Avg.	2.525	2.522	0.003	2.524	2.518	0.006	2.529	2.526	0.003

Notes :

1. Before, After, and Difference refer to Before dry-back, After dry-back, and the difference of the two.

TABLE 6 ANOVA for Rice Gravity Differences Using Aggregate RB

Source	df	SS	MS	F	F _{Crit.}
Total	26	0.00026674	-	-	-
Res. Pr. (A)	2	0.00010496	0.00005248	23.09	3.63 *
Vac. Time (B)	2	0.00000230	0.00000115	0.51	3.63
AxB	4	0.00011215	0.00002804	12.33	3.01 *
Blocks	2	0.00001096	-	-	-
Error	16	0.00003637	0.00000227	-	-

* Significant at $\alpha = 0.05$.

TABLE 7 ANOVA for Rice Gravity Values Before Running Supplementary Procedure Using Aggregate RB

Source	df	SS	MS	F	F _{Crit.}
Total	26	0.00055000	-	-	-
Res. Pr. (A)	2	0.00005267	0.00002633	2.92	3.63
Vac. Time (B)	2	0.00021667	0.00010833	12.00	3.63 *
AxB	4	0.00009733	0.00002433	2.70	3.01
Blocks	2	0.00003889	-	-	-
Error	16	0.00014444	0.00000903	-	-

* Significant at $\alpha = 0.05$.

The factor level 30 mm Hg for Factor A (residual pressure) appears to be supported on the basis of interaction $A \times B$ as well. For Factor B, however, we could select any level. The optimal levels based on the differences, therefore, are

- Residual pressure—30 mm Hg, and
- Vacuuming time—Any level.

Values Before Running Supplementary Procedure

Here only Factor B (vacuuming time) is found to be significant at $\alpha = 0.05$. The objective in this case was to maximize the

value of the theoretical maximum specific gravity before running the supplementary procedure or to eliminate the need for running the supplementary procedure, which is time-consuming and prone to testing errors. To decide the optimal levels, the cumulative averages for Factor B are reported as follows:

Level (min)	Cumulative Average
5	2.525
10	2.526
15	2.531

The optimal levels of factors are selected as

- Residual pressure—Any level, and
- Vacuuming time—15 min.

Combining the results then, the optimal levels of factors for Phase 2 are

- Residual pressure—30 mm Hg, and
- Vacuuming time—15 min.

Finally, as a result of the experiments conducted during Phases 1 and 2 of this study, the following levels of factors should be used when employing the Rice method to determine the theoretical maximum specific gravity of HMA mixtures:

- Temperature—77°F,
- Residual pressure—30 mm Hg, and
- Vacuuming time—15 min.

The use of these optimal levels of factors becomes even more important when one is dealing with absorptive aggregates, which tend to absorb water during the vacuuming phase and thus require a supplementary procedure to correct for this. The use of these levels will minimize the difference between the values of the theoretical maximum specific gravity obtained before and after running the supplementary procedure and may even eliminate the use of the supplementary

procedure for absorptive aggregates if these are adequately coated.

The current ASTM D2041 does specify the temperature to be 77°F. However, there is no restriction on the lower limit of the residual pressure (or, in other words, the upper limit of the vacuum level), which is important while one is dealing with highly absorptive aggregates. This has probably led to the poor reproducibility of the test method.

One more conclusion that has emerged from this study is that it is not necessary to make the water boil during vacuuming phase in order to get all the air out of the sample. This is concluded from the fact that the interaction of temperature and residual pressure was not found to be significant.

CONCLUSIONS AND RECOMMENDATIONS

On the basis of data obtained using two different aggregates and one asphalt cement, the following conclusions are drawn and recommendations made:

1. The following optimum levels of the factors temperature, residual pressure, and vacuuming time are recommended for use when using the Rice method for determining the theoretical maximum specific gravity of an HMA mixture:

- Temperature—77°F,
- Residual pressure—30 mm Hg, and
- Vacuuming time—15 min.

The use of these levels becomes even more important when one deals with absorptive aggregates, which tend to absorb water during the vacuuming phase and require the use of supplementary (dry-back) procedure to correct for the amount of water absorbed. Use of these optimum levels will minimize the difference between the values of the theoretical maximum specific gravity obtained before and after running the supplementary procedure and may even eliminate the use of the supplementary procedure for absorptive aggregates if the aggregate is adequately coated. Use of these same levels by all laboratories is also expected to improve the reproducibility of the Rice method.

2. It is not necessary to make the water boil during vacuuming phase of the Rice method in order to get all the air out of the sample. In fact, the use of lower partial vacuum (30 mm Hg residual pressure instead of 23 mm Hg, as required for boiling at 77°F) is even more important when aggregates of absorptive nature are handled.

3. Proposed modifications to the ASTM D2041 in view of these conclusions and revisions to the procedure are indicated in the following section. Curing of the HMA mix at 290°F for 4 hr before running the Rice method is based on previous research by the authors under SHRP Project A-003B. It should be noted that the proposed modifications are based on data from two mixes only. A multilaboratory round-robin study involving several mixes is recommended.

ADDITIONS AND REVISIONS TO ASTM D2041-90

The proposed revisions and additions to ASTM D2041-90 (Theoretical Maximum Specific Gravity of Bituminous Paving Mixtures) based on this study are given.

Section 6.1.2

• *Old*: The vacuum container size depends on the minimum sample size requirements given in 8.2. Avoid using a small sample in a large container.

• *New*: The vacuum container size depends on the minimum sample size requirements given in 7.2. Avoid using a small sample in a large container.

New Section and Note

• 7.3 For mix design and production control purposes, the sample should be cured in an oven at 290°F for at least 4 hr.

• Note 5: Curing in the oven at the specified temperature is especially important when absorptive aggregates are used. This will ensure the computation of realistic values for the amount of asphalt absorbed by the aggregate and void properties of the mix.

• Note 6: Curing of the mix as required in 7.3 may not be necessary during production if the mix is stored in a surge or storage silo for at least 3 hr. Curing time can also be reduced or eliminated during production if it can be demonstrated through a series of tests conducted after 0, 1, 2, 3, and 4 hr curing time.

Note 5

Re-number existing Note 5 to Note 7.

Section 9.4 and New Note

• *Old*: Remove air trapped in the sample by applying gradually increased vacuum until the residual pressure manometer reads 30 mm of Hg or less. Maintain this residual pressure for 5 to 15 min. Agitate the container. . . .

• *New*: Remove air trapped in the sample by applying gradually increased vacuum until the residual pressure manometer reads 30 mm of Hg. Maintain this residual pressure within ± 1 mm Hg for 15 min. Agitate the container. . . .

• Note 8: It has been found that by using this combination of residual pressure (30 mm Hg) and vacuuming time (15 min), the need for running the supplementary procedure (Section 11) can generally be avoided when testing a thoroughly coated mix.

Note 6

Delete existing Note 6.

Notes 7, 8, and 9

Re-number existing Notes 7, 8, and 9 to Notes 9, 10, and 11.

Section 13

Change all references to "Section 9" in the precision statements to "Section 11."

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The opinions, findings, and conclusions expressed here are those of the authors and not necessarily those of SHRP, NCAT, or Auburn University.