

Asphalt Content by Ignition Round Robin Study

E. R. Brown

and

Stuart Mager

National Center for Asphalt Technology

Auburn University

Asphalt Content by Ignition Round Robin Study

by

E. R. Brown¹ and Stuart Mager²

ABSTRACT

The National Center for Asphalt Technology (NCAT) has developed a test method to determine the asphalt content of hot mix asphalt (HMA) mixtures by ignition. In the ignition method, a HMA sample is subjected to 538°C (1000°F) in a furnace to ignite and burn the asphalt cement from the aggregate. The difference in weight of the sample before and after ignition is used to determine the asphalt content of the mixture. The aggregate recovered after ignition testing may then be used for gradation analysis.

A round robin study was completed by NCAT to determine the accuracy and precision of the ignition method. This paper discusses the round robin test program and the accuracy and precision values determined for the measured asphalt content and gradation by the ignition method. Equipment developed for the procedure was purchased by NCAT with funds provided by the Federal Highway Administration (FHWA), National Asphalt Pavement Association (NAPA), NAPA Research and Education Foundation, and the Alabama Department of Transportation (ADOT). The equipment was provided along with laboratory

¹Director, National Center for Asphalt Technology, and ²Graduate Research Assistant, Auburn University.

prepared HMA samples to 12 participating laboratories throughout the U. S. Four replicates of four HMA mixtures containing different aggregates types, gradations and asphalt contents were provided for testing.

The results of the round robin study show that the ignition method can accurately measure the AC content of HMA mixtures with greater precision than solvent extraction methods without significantly affecting the gradation of the aggregate. This test method has shown excellent potential for replacing existing test methods for measuring asphalt content.

Asphalt Content by Ignition

Round Robin Study

I. INTRODUCTION

1.1 Background

The National Center for Asphalt Technology (NCAT) has developed a test procedure to measure asphalt content of hot mix asphalt (HMA) mixtures by ignition. This test burns the asphalt cement from the mixture and provides information needed to determine the asphalt content. Since this is a new test procedure, the accuracy and precision need to be determined.

1.2 Objective

The objective of this study was to determine the accuracy and precision values for asphalt content determination by the ignition test method. The study was to also determine the accuracy and precision values for the measured gradation of the recovered aggregate determined after the asphalt was removed from the mix.

1.3 Scope

The round robin study was performed according to ASTM C802 standard practice for conducting an interlaboratory test program to determine the precision of test methods for construction materials. A minimum of ten participating laboratories is recommended by ASTM. NCAT chose thirteen laboratories around the United States which included State

Departments of Transportation, HMA producers and the Federal Highway Administration (FHWA).

Each laboratory was supplied with one NCAT Asphalt Content Tester and two sets of sample baskets for testing. Four different HMA mixture types consisting of different aggregates, gradations, and asphalt contents were prepared at NCAT and sent to the participant laboratories for asphalt content determination and gradation analysis. The laboratories had no knowledge of the asphalt content nor gradation of the HMA samples. A test procedure, instructions and summary data sheets were sent along with the test samples to each laboratory. Each laboratory was asked to follow the test procedure provided and report the results back to NCAT for analysis.

The collected interlaboratory test data was analyzed and the accuracy and precision values for the HMA properties were calculated using appropriate statistical methods.

II. INTERLABORATORY TEST PROGRAM

2.1 Material and Sample Preparation

HMA mixtures were prepared with four different types of aggregate and one type of asphalt cement. The materials used to prepare the HMA test samples are shown in Table 1. Calibration samples also were made with the same four aggregate types, but were not mixed with asphalt cement. HMA samples were prepared with a known asphalt cement content and gradation as shown in Table 2. Four different dense gradations and three different asphalt contents ranging from 5.0 to 6.0 percent were used in preparing the four HMA mixture types.

Table 1. Materials Used for Preparing Test Samples

| | Material |
|----------------------|-----------|
| Aggregate #1 | Gravel |
| Aggregate #2 | Granite |
| Aggregate #3 | Limestone |
| Aggregate #4 | Traprock |
| Asphalt Cement Grade | AC-20 |

Each aggregate type was oven dried and then separated into individual sieve sizes to meet the desired gradation. The approximate batch weight of each sample was 1200 grams. Samples of each mix type were batched and a washed-sieve analysis was performed according to ASTM C136 and C117 to determine the true gradation. The average of these values provides the best measure of the true gradation of each mix type. These average gradations are shown in Table 2.

After the aggregate was batched, HMA samples were prepared by mixing the aggregate with the required amount of asphalt cement. The mixing equipment was conditioned with a “butter” mix of aggregate and asphalt cement before mixing of the test samples. The “butter” mix uniformly coats the mixing equipment so that subsequent samples can be more completely removed from the equipment after mixing.

Table 2. Aggregate Type, Aggregate Gradation and Asphalt Content for Mixes Used in Round Robin Study

| Aggregate | Gravel | Granite | Limestone | Traprock |
|--------------------------------|-----------------|---------|-----------|----------|
| AC Content, % | 6.00* | 6.00'' | 5.00' | 5.50' |
| Aggregate Gradation Sieve Size | Percent Passing | | | |
| 19 mm (3/4 inch) | 100.0 | 100.0 | 100.0 | 100.0 |
| 12.5 mm (1/2 inch) | 97.3 | 97.7 | 97.4 | 97.0 |
| 9.5 mm (3/8 inch) | 88.5 | 85.8 | 85.6 | 83.5 |
| 4.75 mm (No. 4) | 71.6 | 66.8 | 61.4 | 57.0 |
| 2.36 mm (No. 8) | 50.6 | 50.1 | 43.5 | 39.9 |
| 1.18mm (No. 16) | 35.7 | 36.0 | 30.8 | 29.1 |
| 600 microns (No. 30) | 25.1 | 25.3 | 22.0 | 20.2 |
| 300 microns (No. 50) | 15.3 | 16.1 | 14.6 | 13.6 |
| 150 microns (No. 100) | 8.9 | 10.9 | 9.3 | 8.4 |
| 75 microns (No. 200) | 6.0 | 7.7 | 6.7 | 5.3 |

* Aggregate was also provided with the same gradation, but with no asphalt cement for calibration.

2.2 Sample Packaging

A container was needed for shipping the HMA samples to each laboratory that could withstand heat from the hot samples and that would allow easy removal of the sample at each of the participating laboratories. Different types of containers were obtained and evaluated. A cardboard box with a wax like coating on the inside was selected and obtained from the Menasha Corporation in Menasha, Wisconsin for sending the samples to each of the round robin participants. This container could be heated without damage and the coating minimized the amount of the sample remaining in the container. After samples were prepared, they were placed immediately in the container for shipping. Calibration samples (aggregate only) were placed in plastic “ziplock” bags for shipping. The calibration sample could then be removed from the bag and placed in a metal bowl for heating in an oven prior to ignition testing. Before sending the samples to each laboratory, each sample was weighed to confirm that correct total weight was provided in each container.

The samples were labeled according to the type of mix and were given a number representing either a calibration sample or an HMA sample. Mix types 1, 2, 3 and 4 were labeled A, B, C and D respectively. Sample numbers 1-4 represented HMA samples and sample numbers 5-8 represented calibration samples. Figure 1 shows the shipping carton , containing a HMA sample.

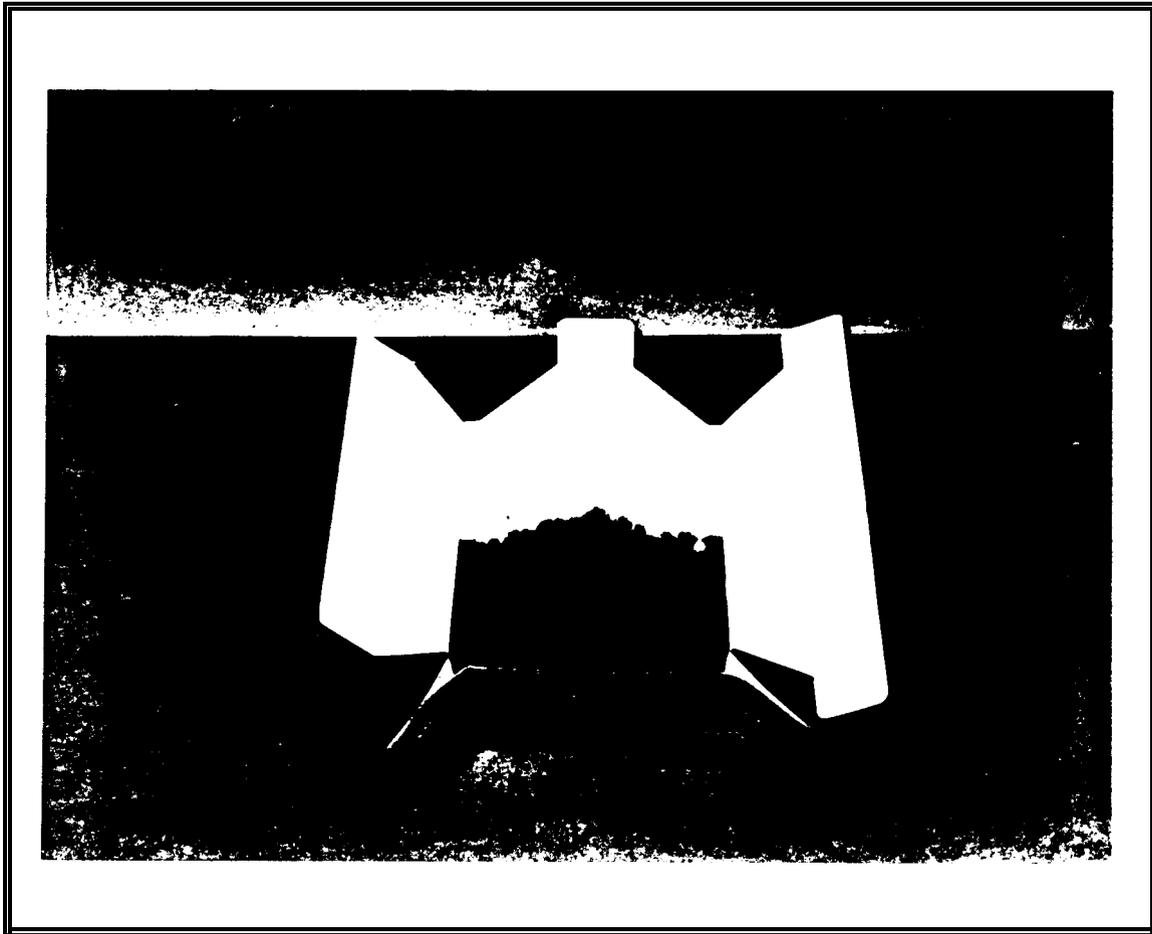


Figure 1. Shipping Carton Containing HMA Test Sample

2.3 Equipment

Each laboratory was supplied with one Asphalt Content Tester and two sets of sample baskets. The test equipment and sample baskets are shown in Figures 2 and 3.

The Asphalt Content Tester consists of a muffle furnace with a built-in scale that continuously weighs the sample during the test. The unit measures and displays the weight loss of the sample with a digital readout. A built-in printer prints the asphalt content on

completion of the test. The unit has a filter that reduces the smoke produced during ignition of the asphalt cement to acceptable levels. The operator is only required to input the weight of the sample, the required test temperature and the calibration factor before beginning the test.

The sample baskets consist of two stainless steel No. 8 mesh trays which are stacked on top of each other and placed on top of a flat stainless steel catch pan. The HMA test sample or calibration sample is equally divided into two portions and placed into the two mesh trays. The mesh trays are fastened to the catch pan with a safety strap and are inserted into the furnace for testing.

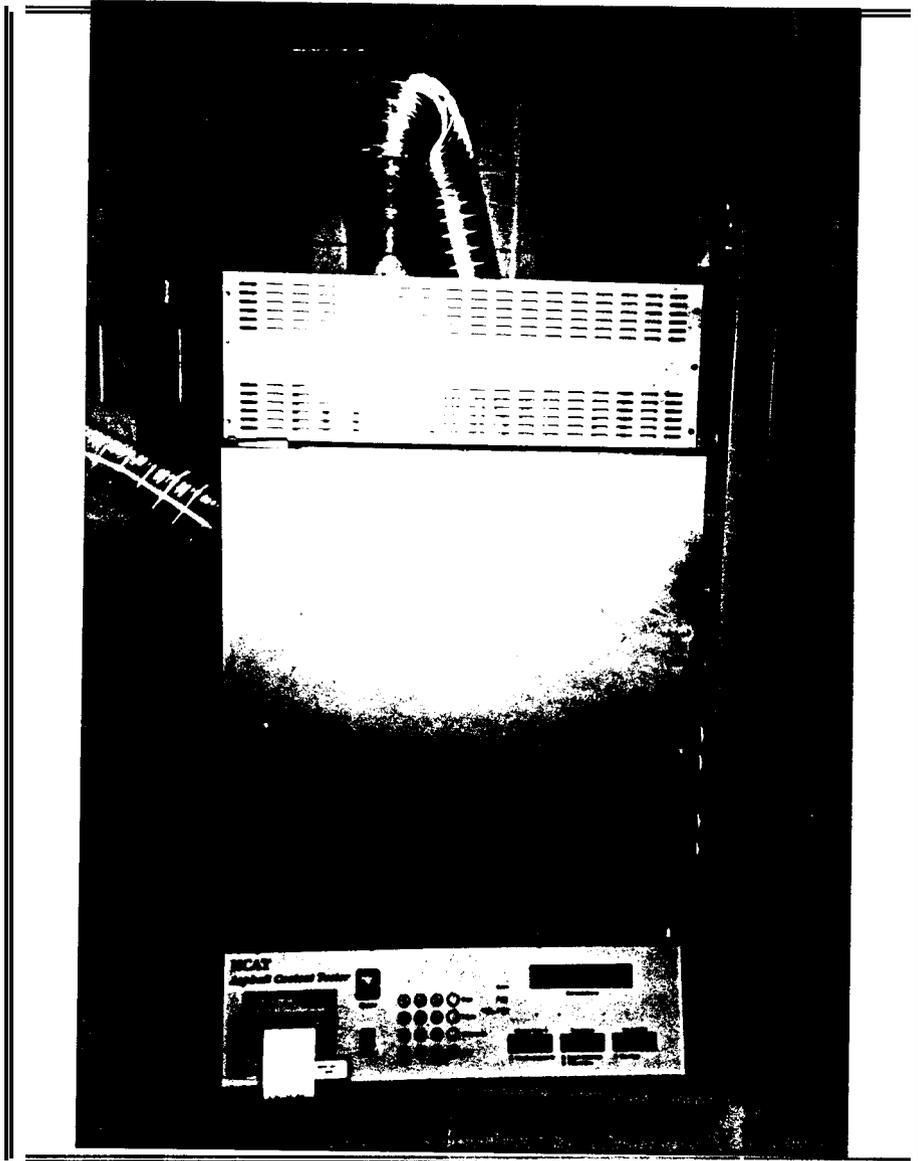


Figure 2. Asphalt Content Tester

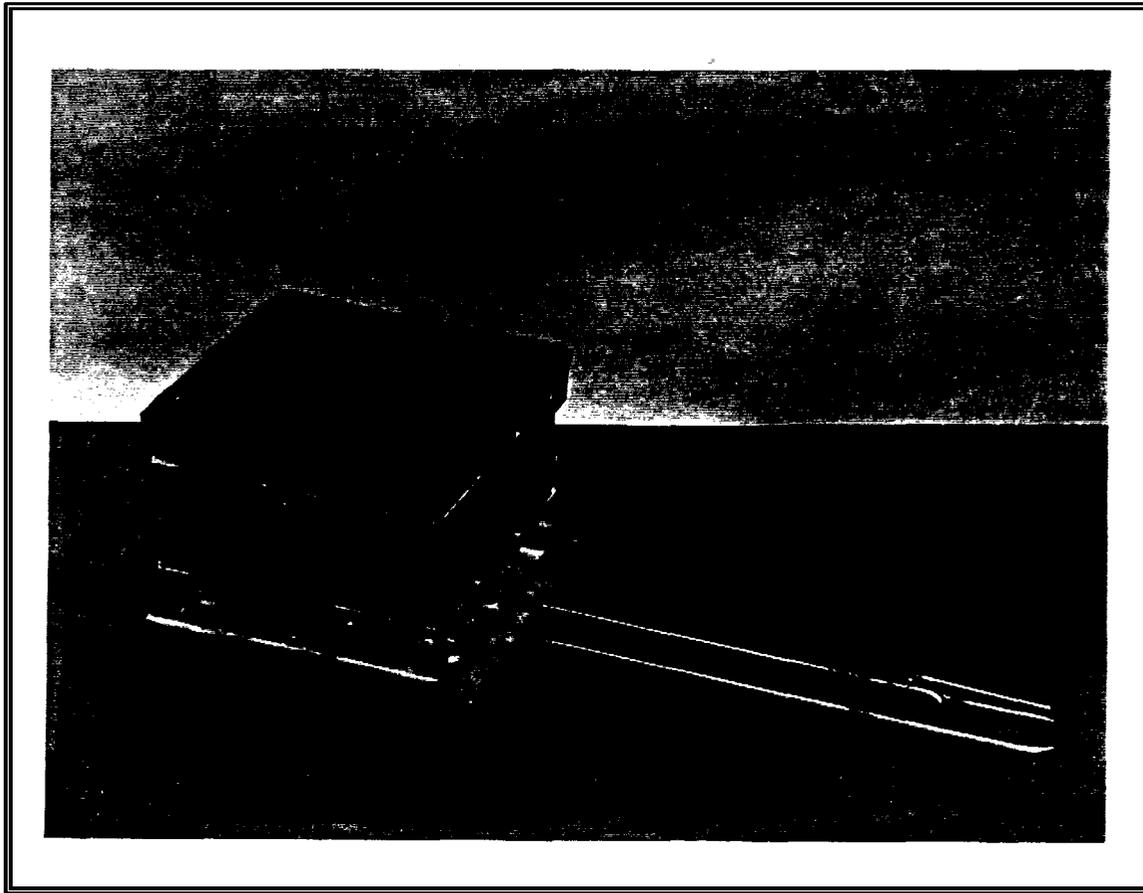


Figure 3. Test Sample Baskets

2.4 Test Procedure

A test procedure for the interlaboratory test program was developed and provided to each participant. The procedure was developed for use solely with the test equipment used in this study. The test procedure was written so that the operator could perform the test easily in a step by step process and so that every laboratory would perform the test in an identical manner. Each laboratory was provided with two extra HMA samples to familiarize the operator with the test procedure and to ensure that the equipment was functioning properly before testing of the round robin samples was initiated.

Research at NCAT has shown that the optimum temperature for ignition testing of most aggregates is 538 °C (1000°F). Higher temperatures increase the amount of aggregate mass loss for some aggregates and lower test temperatures may excessively increase the test time. Therefore, the test procedure for the round robin study utilized a test temperature of 538°C(1000°F) for testing of HMA samples. The samples were burned until the measured weight loss did not exceed 0.1 gram for three consecutive minutes. The time required to achieve a constant weight was approximately 30-40 minutes. During testing of HMA samples, the temperature inside the furnace increased approximately 40°C(72°F) to 578°C(1072°F) once the asphalt cement ignited.

Generally, most aggregates will experience some mass loss (usually less than 0.4 percent) due to ignition testing. As a result, the measured weight loss of HMA samples will consist of the weight loss of the asphalt cement and the weight loss of the aggregate. Therefore, to optimize accuracy, calibration samples (aggregate only) were burned to determine the amount of aggregate mass loss of each aggregate type. In order to subject the calibration samples to the same test conditions as the HMA samples, the test procedure called for burning calibration samples for 40 minutes at a test temperature of 578 °C(1072°F).

2.5 Interlaboratory Testing Results

The results of the round robin study were collected from each laboratory and analyzed for accuracy and precision according to ASTM C802 and ASTM C670. Data from twelve laboratories was used to determine the precision and accuracy of the test method. One of the thirteen laboratories did not report any results. The measured AC content, percent passing the 4.75 mm (No. 4) sieve and percent passing the 75 micron (No. 200) sieve for each

laboratory are shown in Tables 3 through 5. A total of 15 samples from 8 laboratories were damaged prior to or during the test. ASTM C802 recommends making additional samples and collecting the missing data if the number of missing data from all the laboratories exceeded 1% of the total. Since this number (7.8% of the 192 samples sent to round robin participants) exceeds the minimum recommended percentage of 1 % according to ASTM C802, additional samples were prepared and sent to these laboratories. Therefore, 15 replacement samples were sent to the respective laboratories to test. The original damaged samples were discarded and the replacement samples were tested and used in the analysis for accuracy and precision.

Table 3 shows that all of the measured asphalt contents for the four repetitions of the four mixes are very close to the true asphalt content of 6.0 percent. In fact, the measured asphalt content of the 192 samples that deviates the most from the true asphalt content is only off by 0.23 *o/o*. No sample was discarded as an outlier.

Table 4 shows that the measured percentage passing the 4.75 mm(No. 4) sieve for each test is very close to the actual percentage passing as determined before the ignition test. The worst test result deviated 1.2 % from the actual percentage passing. This amount of variability is acceptable.

Table 5 shows that there is more variability in the, percentage passing the 75 micron(No. 200) sieve than there was for the percentage passing the 4.75 mm(No. 4) sieve. The worst test result here deviated by 2.9 percent from the true percent passing. It does

Table3. Measured AC Content

| Lab | | Measured AC Content, b | | | |
|---------|---|------------------------|-------|-------|-------|
| | | Mix 1 | Mix 2 | Mix 3 | Mix 4 |
| 1 | 1 | 5.96 | 6.02 | 5.03 | 5.13 |
| 1 | 2 | 5.98 | 6.03 | 5.05 | 5.49 |
| 1 | 3 | 6.01 | 6.01 | 5.05 | 5.50 |
| 1 | 4 | 5.99 | 6.04 | 5.07 | 5.54 |
| m | 4 | 5.99 | 6.00 | 4.88 | 5.51 |
| 2 | i | 5.93 | 6.02 | 4.91 | 5.49 |
| 2 | 3 | 5.94 | 6.01 | 4.90 | 5.58 |
| 2 | 4 | 5.94 | 5.95 | 4.93 | 5.58 |
| 3 | 1 | 5.98 | 5.87 | 4.97 | 5.50 |
| 3 | 2 | 6.01 | 5.96 | 4.88 | 5.56 |
| 3 | 3 | 6.00 | 5.97 | 5.00 | 5.54 |
| 3 | 4 | 5.94 | 6.00 | 4.94 | 5.51 |
| 4 | 1 | 5.97 | 5.98 | 4.87 | 5.52 |
| 4 | 2 | 6.01 | 5.99 | 5.03 | 5.54 |
| 4 | 3 | 6.02 | 5.99 | 4.99 | 5.54 |
| 4 | 4 | 5.99 | 6.00 | 4.97 | 5.48 |
| 5 | 1 | 5.98 | 5.98 | 5.00 | 5.43 |
| 5 | 2 | 5.97 | 6.00 | 4.89 | 5.40 |
| 5 | 3 | 5.99 | 6.01 | 4.99 | 5.40 |
| 5 | 4 | 6.00 | 5.99 | 4.89 | 5.41 |
| 6 | 1 | 5.93 | 6.01 | 4.98 | 5.57 |
| 6 | 2 | 6.01 | 5.95 | 4.98 | 5.54 |
| 6 | 3 | 5.99 | 5.97 | 4.99 | 5.52 |
| 6 | 4 | 5.96 | 5.99 | 5.01 | 5.55 |
| 7 | 1 | 5.95 | 5.91 | 4.90 | 5.47 |
| 7 | 2 | 5.97 | 5.95 | 4.90 | 5.49 |
| 7 | 3 | 6.01 | 5.92 | 4.90 | 5.38 |
| 7 | 4 | 5.95 | 5.97 | 4.91 | 5.39 |
| 8 | 1 | 5.84 | 6.00 | 4.89 | 5.50 |
| 8 | 2 | 5.90 | 6.02 | 5.04 | 5.54 |
| 8 | 3 | 6.00 | 5.98 | 5.03 | 5.60 |
| 8 | 4 | 5.92 | 5.97 | 4.88 | 5.60 |
| 9 | 1 | 6.06 | 6.05 | 5.00 | 5.70 |
| 9 | 2 | 6.07 | 6.09 | 4.97 | 5.64 |
| 9 | 3 | 6.10 | 6.09 | 5.02 | 5.58 |
| 9 | 4 | 6.03 | 6.10 | 5.01 | 5.52 |
| 10 | 1 | 5.98 | 6.04 | 4.95 | 5.59 |
| 10 | 2 | 5.92 | 5.99 | 5.03 | 5.59 |
| 10 | 3 | 5.99 | 5.99 | 5.04 | 5.64 |
| 10 | 4 | 5.99 | 5.01 | 5.03 | 5.63 |
| 11 | 1 | 6.02 | 5.93 | 5.01 | 5.60 |
| 11 | 2 | 5.97 | 5.95 | 5.11 | 5.54 |
| 11 | 3 | 5.99 | 6.02 | 4.95 | 5.52 |
| 11 | 4 | 5.83 | 6.01 | 4.94 | 5.49 |
| 12 | 1 | 6.00 | 5.95 | 4.93 | 5.45 |
| 12 | 2 | 6.00 | 5.99 | 4.98 | 5.47 |
| 12 | 3 | 5.95 | 5.96 | 4.94 | 5.49 |
| 12 | 4 | 6.00 | 6.02 | 4.93 | 5.49 |
| Average | | 5.98 | 5.99 | 4.97 | 5.53 |
| True | | 6.00 | 6.00 | 5.00 | 3.30 |

Table 4. Measured Percent Passing 4.75 mm (No. 4) Sieve

| Lab | Sample | Percent Passing, % | | | |
|---------|--------|--------------------|-------|-------|-------|
| | | Mix 1 | Mix 2 | Mix 3 | Mix 4 |
| 1 | 1 | 71.5 | 66.4 | 61.2 | 56.5 |
| 1 | 2 | 71.4 | 66.4 | 61.3 | 56.4 |
| 1 | 3 | 71.4 | 66.2 | 61.0 | 56.6 |
| 1 | 4 | 71.1 | 66.4 | 61.1 | 56.5 |
| 2 | 1 | 71.0 | 66.0 | 61.0 | 56.0 |
| 2 | 2 | 71.0 | 67.0 | 61.0 | 56.0 |
| 2 | 3 | 71.0 | 66.0 | 61.0 | 56.0 |
| 2 | 4 | 71.0 | 66.0 | 62.0 | 56.0 |
| 3 | 1 | 71.9 | 67.0 | 61.2 | 56.7 |
| 3 | 2 | 71.6 | 67.1 | 61.6 | 57.7 |
| 3 | 3 | 71.7 | 67.1 | 61.5 | 56.6 |
| 3 | 4 | 71.6 | 67.5 | 61.7 | 56.8 |
| 4 | 1 | 71.5 | 66.6 | 61.3 | 56.8 |
| 4 | 2 | 71.9 | 66.3 | 61.4 | 56.7 |
| 4 | 3 | 71.7 | 66.7 | 61.9 | 56.6 |
| 4 | 4 | 71.8 | 66.5 | 61.6 | 56.2 |
| 5 | 1 | 71.3 | 66.7 | 61.3 | 56.4 |
| 5 | 2 | 71.4 | 66.7 | 61.2 | 56.8 |
| 5 | 3 | 71.4 | 66.7 | 61.2 | 56.8 |
| 5 | A | 71.5 | 66.7 | 61.2 | 56.3 |
| 6 | 1 | 71.0 | 67.2 | 61.4 | 56.6 |
| 6 | 2 | 71.9 | 67.2 | 61.5 | 56.3 |
| 6 | 3 | 71.8 | 66.7 | 61.4 | 56.5 |
| 6 | 4 | 71.8 | 66.8 | 61.4 | 56.5 |
| 7 | 1 | 71.8 | 66.4 | 61.2 | 56.4 |
| 7 | 2 | 71.5 | 66.7 | 61.0 | 57.1 |
| 7 | 3 | 71.2 | 65.8 | 61.0 | 56.2 |
| 7 | 4 | 71.7 | 66.8 | 61.1 | 56.6 |
| 8 | 1 | 71.0 | 66.3 | 60.9 | 56.3 |
| 8 | 2 | 70.0 | 66.7 | 61.4 | 56.8 |
| 8 | 3 | 71.0 | 66.2 | 61.3 | 56.5 |
| 8 | A | 71.1 | 66.2 | 61.1 | 56.3 |
| 9 | 1 | 71.5 | 67.2 | 61.3 | 56.7 |
| 9 | 2 | 71.3 | 66.9 | 61.6 | 56.4 |
| 9 | 3 | 71.4 | 67.0 | 61.4 | 56.2 |
| 9 | 4 | 71.1 | 66.9 | 61.2 | 56.2 |
| 9 | t | 71.6 | 66.2 | 61.2 | 56.2 |
| 10 | 2 | 71.8 | 66.5 | 61.4 | 57.0 |
| 10 | 3 | 71.4 | 66.2 | 61.6 | 56.9 |
| 10 | 4 | 71.2 | 66.3 | 61.2 | 56.4 |
| 11 | 1 | 71.3 | 66.3 | 60.9 | 56.1 |
| 11 | 2 | 71.1 | 66.5 | 61.1 | 56.8 |
| 11 | 3 | 71.1 | 66.5 | 61.1 | 56.2 |
| 11 | 4 | 71.5 | 66.3 | 61.6 | 56.8 |
| 12 | 1 | 71.6 | 67.1 | 61.3 | 57.2 |
| 12 | 2 | 72.0 | 67.4 | 61.5 | 57.0 |
| 12 | 3 | 71.2 | 67.4 | 62.5 | 58.2 |
| 12 | 4 | 71.6 | 67.0 | 61.7 | 56.5 |
| Average | | 71.5 | 66.6 | 61.4 | 56.6 |
| Control | | 71.6 | 66.8 | 61.4 | 57.0 |

Table 5. Measured Percent Passing 75 micron (No. 200) Sieve

| Lab | Sample | Percent | | Passing, % | |
|---------|--------|---------|-------|------------|-------|
| | | Mix 1 | Mix 2 | Mix 3 | Mix 4 |
| 1 | 1 | 59 | 80 | 7.1 | 5.5 |
| 1 | 2 | 5.9 | 8.1 | 7.3 | 5.4 |
| 1 | 3 | 6.0 | 7.8 | 7.2 | 5.2 |
| 1 | 4 | 5.9 | 7.6 | 7.3 | 5.3 |
| 2 | 1 | 5.8 | 7.6 | 6.8 | 5.0 |
| 2 | 2 | 5.0 | 8.0 | 8.0 | 5.0 |
| 2 | 3 | 5.8 | 7.5 | 6.6 | 4.0 |
| 2 | 4 | 5.8 | 7.9 | 7.5 | 5.0 |
| 3 | 1 | 6.6 | 8.4 | 7.1 | 5.5 |
| 3 | 2 | 6.5 | 8.8 | 7.4 | 5.5 |
| 3 | 3 | 6.5 | 8.7 | 8.4 | 5.3 |
| 3 | 4 | 6.5 | 8.5 | 8.0 | 5.5 |
| 4 | 1 | 5.3 | 8.2 | 6.9 | 5.4 |
| 4 | 2 | 5.5 | 8.1 | 6.9 | 4.8 |
| 4 | 3 | 5.8 | 8.3 | 8.1 | 4.7 |
| 4 | 4 | 5.8 | 8.3 | 6.9 | 4.8 |
| 5 | 1 | 5.6 | 8.5 | 7.1 | 5.5 |
| 5 | 2 | 4.9 | 8.1 | 6.9 | 5.5 |
| 5 | 3 | 5.9 | 7.9 | 6.7 | 5.5 |
| 5 | 4 | 5.5 | 8.0 | 7.0 | 5.2 |
| 6 | 1 | 5.8 | 8.0 | 6.8 | 5.2 |
| 6 | 2 | 6.4 | 7.5 | 8.0 | 5.3 |
| 6 | 3 | 6.4 | 7.7 | 8.1 | 5.3 |
| 6 | 4 | 5.5 | 8.1 | 7.0 | 5.3 |
| 7 | 1 | 6.2 | 8.0 | 7.7 | 5.2 |
| 7 | 2 | 5.9 | 8.1 | 7.0 | 5.1 |
| 7 | 3 | 5.9 | 7.7 | 7.8 | 5.3 |
| 7 | 4 | 6.3 | 7.5 | 6.7 | 4.9 |
| 8 | 1 | 4.5 | 6.5 | 7.1 | 4.4 |
| 8 | 2 | 4.7 | 7.1 | 6.4 | 4.6 |
| 8 | 3 | 5.0 | 6.5 | 5.8 | 3.7 |
| 8 | 4 | 4.8 | 7.0 | 6.0 | 3.9 |
| 9 | 1 | 5.7 | 8.0 | 8.0 | 5.3 |
| 9 | 2 | 5.9 | 8.2 | 8.0 | 5.1 |
| 9 | 3 | 5.5 | 7.7 | 7.0 | 5.3 |
| 9 | 4 | 6.3 | 8.2 | 6.8 | 4.9 |
| 10 | 1 | 5.4 | 7.6 | 7.2 | 5.1 |
| 10 | 2 | 5.2 | 7.6 | 7.4 | 5.1 |
| 10 | 3 | 5.3 | 7.1 | 7.2 | 4.9 |
| 10 | 4 | 6.0 | 7.5 | 7.4 | 5.0 |
| 11 | 1 | 3.2 | 4.8 | 7.5 | 4.7 |
| 11 | 2 | 3.2 | 5.0 | 6.6 | 4.7 |
| 11 | 3 | 3.2 | 8.5 | 6.7 | 5.2 |
| 11 | 4 | 6.1 | 7.3 | 6.4 | 5.0 |
| 12 | 1 | 6.5 | 8.4 | 7.0 | 5.1 |
| 12 | 2 | 6.0 | 7.9 | 7.3 | 5.6 |
| 12 | 3 | 6.0 | 7.7 | 8.3 | 5.6 |
| 12 | 4 | 5.7 | 8.2 | 7.3 | 5.0 |
| Average | | 5.6 | 7.7 | 7.2 | 5.1 |
| control | | 6.0 | 7.7 | 6.7 | 5.3 |

appear for laboratory 11 that 5 of the 16 test results are in error. Even though these 5 tests appear to be outliers, they were used to calculate precision and accuracy. If the five “bad” tests from laboratory 11 are excluded then the worst test result of the remaining 187 tests deviates from the actual gradation by 1.7%.

2.6 Statistical Analysis

2.6.1 Determination of Within and Between Laboratory Variances

The within and between laboratory variances were determined according to ASTM C802. The test for outliers and homogeneity of variance was not performed on the data for this study. All data was included in the analysis. None was discarded as outliers. The components of variance, variances and standard deviations were calculated according to ASTM C802.

2.6.2 Measured AC Content

Each laboratory conducted ignition testing for 16 HMA samples. Four replicates of four mixtures were tested for AC content determination. The measured asphalt contents for mix #1 ranged from 5.83 to 6.07 percent. Mix #2 measured AC contents ranged from 5.87 to 6.10 percent. The true AC content for mix #1 and #2 was 6.00 percent. The true AC contents for mix #3 and #4 were 5.00 and 5.50 percent respectively. The measured AC contents for mix #3 ranged from 4.87 to 5.11 percent. Mix #4 measured AC contents ranged from 5.38 to 5.73 percent. The maximum difference between the true AC content and the measured AC content for the four mixes was 0.23 percent for mix 4. So for a total of 192 tests, the worst test result was 0.23 percent from the “true” asphalt content.

2.6.2 Accuracy of Measured AC Content

The measured AC contents from each laboratory were averaged for each mixture type. The average measured AC contents are shown in Table 6. Each number shown is the average of 48 test results with 12 laboratories performing tests on 4 replicates of each mixture type.

As shown in Table 6, the deviation of the measured AC content from the true AC content ranged from -0.03 to +0.03 percent. The overall average deviation of the measured AC content for the 192 samples tested was -0.02 percent. The low bias measured indicates that the AC content of HMA mixtures can be obtained with a high degree of accuracy using the ignition method.

Table 6. Accuracy of Ignition Test for Asphalt Content

| Mix | “True” AC Content, % | Average Measured AC Content, % | Bias, % |
|-----|-------------------------|--------------------------------------|---------|
| 1 | 6.00 | 5.98 | -0.02 |
| 2 | 6.00 | 5.99 | -0.01 |
| 3 | 5.00 | 4.97 | -0.03 |
| 4 | 5.50 | 5.53 | 0.03 |

2.6.3 Precision of Measured AC Content

The within laboratory and the between laboratory standard deviations for the measured AC content are shown in Table 7 according to mix type. The symbols W/L and B/L represent the within laboratory and between laboratory components respectively. The within laboratory standard deviation ranged from 0.03 to, 0.05 percent. The overall within laboratory standard

deviation was 0.04 percent. The between laboratory standard deviation ranged from 0.5 to 0.08 percent. The average between laboratory standard deviation was 0.06 percent. The solvent extraction method as specified in ASTM has been shown to have standard deviations for within laboratory and between laboratory of 0.21 and 0.22 percent respectively. Compared to the solvent extraction method, the ignition method has significantly lower standard deviations and therefore provides a higher degree of precision.

Table 7. Components of Variance, Variances and Standard Deviations for Asphalt Content

| Mix | Component of Variance | | Variance | | Standard Deviation | |
|-----|-----------------------|--------|----------|--------|--------------------|--------|
| | W/L | B/L | W/L | B/L | W/L | B/L |
| 1 | 0.0016 | 0.0010 | 0.0016 | 0.0026 | 0.0405 | 0.0513 |
| 2 | 0.0009 | 0.0012 | 0.0009 | 0.0021 | 0.0297 | 0.0460 |
| 3 | 0.0022 | 0.0014 | 0.0022 | 0.0036 | 0.0468 | 0.0595 |
| 4 | 0.0026 | 0.0032 | 0.0026 | 0.0059 | 0.0514 | 0.0766 |

2.6.4 Aggregate Gradation

Testing of aggregates at high temperatures will generally result in aggregate weight loss. The test temperature for ignition testing was 538°C(1000°F). The aggregate was recovered after testing and evaluated to determine if there was a significant change in gradation due to the high test temperature. Accuracy and precision of the percent passing the 4.75 mm and 75 mm sieves was determined. The measured test results are shown in Tables 8 and 9.

2.6.5 Accuracy of Percent Passing the 4.75mm Sieve

The average percent passing the No. 4 sieve for each mix is shown in Table 8. The bias for the four mix types ranged from -0.4 to 0.0 percent. The overall difference in percent passing the No. 4 sieve after testing was -0.02 percent. Since the difference between the true percent passing and the measured percent passing was very low, the percent passing the 4.75 mm sieve can be determined with a high degree of accuracy.

Table 8. Accuracy of Ignition Test for Percent Passing 4.75 mm Sieve

| Mix | “True” Percent Passing 4.75 mm Sieve | Average Measured Percent Passing 4.75 mm Sieve After Ignition Test | Bias, % |
|-----|--|---|---------|
| 1 | 71.6 | 71.5 | -0.1 |
| 2 | 66.8 | 66.6 | -0.2 |
| 3 | 61.4 | 61.4 | 0.0 |
| 4 | 57.0 | 56.6 | -0.4 |

2.6.6 Precision of Percent Passing 4.75 mm Sieve

The within laboratory and between laboratory standard deviations for the percent passing the 4.75 mm sieve are shown in Table 9. The within laboratory standard deviations range from 0.22 to 0.34 percent. The between laboratory standard deviations range from 0.31 to 0.42 percent. The overall within-laboratory and between-laboratory standard deviations were 0.27 and 0.37 percent respectively.

Table 9. Components of Variance, Variances and Standard Deviations for Percent Passing 4.75 mm Sieve

| Mix | Component of Variance | | Variance | | Standard Deviation | |
|-----|-----------------------|--------|----------|--------|--------------------|--------|
| | W/L | B/L | W/L | B/L | W/L | B/L |
| 1 | 0.0496 | 0.0592 | 0.0496 | 0.1088 | 0.2227 | 0.3299 |
| 2 | 0.0519 | 0.1021 | 0.0519 | 0.1540 | 0.2278 | 0.3924 |
| 3 | 0.0794 | 0.0161 | 0.0794 | 0.0955 | 0.2817 | 0.3090 |
| 4 | 0.1183 | 0.0621 | 0.1183 | 0.1804 | 0.3440 | 0.4247 |

2.6.7 Accuracy of Percent Passing the 75 mm Sieve

The deviation of the measured percent passing the 75 mm sieve from the true percent passing for each mix is shown in Table 10. The bias ranges from -0.4 to 0.5 percent. The overall difference was -0.1 percent.

Table 10. Accuracy of Ignition Test for Percent Passing 75 μm Sieve

| Mix | “True” Percent Passing 75 μm Sieve | Average Measured Percent Passing 75 μm Sieve After Ignition Test | Bias, % |
|-----|---|--|---------|
| 1 | 6.0 | 5.6 | -0.4 |
| 2 | 7.7 | 7.7 | 0.0 |
| 3 | 6.7 | 7.2 | 0.5 |
| 4 | 5.3 | 5.1 | -0.2 |

2.6.8 Precision of Percent Passing the 75 μm Sieve

The values for the within laboratory and between laboratory standard deviations are shown in Table 11. The within laboratory standard deviations for the four mix types range from 0.26 to 0.57 percent. The between laboratory standard deviations range from 0.43 to 0.82 percent. The overall within laboratory standard deviation was 0.47. The overall between laboratory standard deviation was 0.65 percent.

Table 11. Components of Variance, Variances and Standard Deviations for Percent Passing 75 μm Sieve

| Mix | Component of Variance | | Variance | | Standard Deviation | |
|-----|-----------------------|--------|----------|--------|--------------------|--------|
| | W/L | B/L | W/L | B/L | W/L | B/L |
| 1 | 0.2623 | 0.4064 | 0.2623 | 0.6687 | 0.5121 | 0.8177 |
| 2 | 0.3269 | 0.2937 | 0.3269 | 0.6205 | 0.5717 | 0.7877 |
| 3 | 0.2667 | 0.0708 | 0.2667 | 0.3375 | 0.5165 | 0.5809 |
| 4 | 0.0689 | 0.1153 | 0.0689 | 0.1842 | 0.2624 | 0.4291 |

2.7 Precision Statement

The precision statement for asphalt content and gradation were written according to ASTM C670. The precision value calculated is the acceptable range of two test results (D2S). The precision statements were not written for each mixture type. They were calculated by taking an average of all four mixtures.

Table 12 shows the precision statement for asphalt content, percent passing the 4.75 mm sieve and percent passing the 75 μm sieve. The within laboratory (0.04) and between laboratory (0.06) standard deviations are much lower than the 0.21 and 0.22 percent standard deviations with the extraction test. There are no typical values for comparison of the variability of gradation since the samples tested in this study were prepared with aggregate that was batched to meet the design gradation.

The precision and bias statement developed here is for 4 aggregates types. These calculated numbers are expected to be applicable for most aggregates. However, there might be aggregates that have not been evaluated that do not do as well in the test. Additional work is needed to verify these precision and bias numbers for a wide range of aggregate types.

Table 12. Precision Statement for Asphalt Content, Percent Passing 4.75 mm Sieve and Percent Passing 75 μ m Sieve Determined by the Ignition Method

| Test Property | Standard Deviation (1S) | | Acceptable Range of Two Test Results (D2S) | |
|--|-------------------------|------|--|------|
| | W/L | B/L | W/L | B/L |
| Asphalt Content | 0.04 | 0.06 | 0.11 | 0.17 |
| Percent Passing 4.75 mm Sieve | 0.27 | 0.37 | 0.8 | 1.1 |
| Percent Passing 75 μ m Sieve | 0.47 | 0.65 | 1.3 | 1.8 |
| Basis of Estimate: 4 replicates 4 materials 12 laboratories | | | | |

III. CONCLUSIONS

3.1 General

Extensive laboratory. research was performed to develop a revised test procedure. After a test procedure was developed, an interlaboratory test program was carried out to determine the precision of the test method.

3.2 Conclusions

Based on the results presented in this report, the following conclusions can be drawn:

1. The ignition method can be used to accurately and quickly determine the AC content and gradation of HMA mixtures.
2. Statistical analysis shows that the ignition method can determine the asphalt content with greater precision than the solvent extraction method. The within laboratory and between laboratory standard deviations for AC content as determined from the interlaboratory test program were 0.04 and 0.06 percent respectively. Comparatively, the within laboratory and between laboratory standard deviations for AC content determination by the solvent extraction method are 0.21 and 0.22 percent respectively.
3. The test procedure is very simple and can be performed in approximately 30 minutes for 1200 gram samples. The operator must only be present to start the test. The operator does not need to be present while the test is in progress.
4. The ignition method is relatively inexpensive. There are no associated costs for disposal of hazardous solvents.

5. The recovered aggregate is free of asphalt cement and can be used for gradation analysis.
6. Smoke produced during ignition testing is reduced to acceptable levels with a filter for use in the field.

REFERENCES

1. Brown, E. R., N. Murphy, and S. Mager, "Historical Development of Asphalt Content Determination by the Ignition Method." *Journal of Asphalt Paving Technologists*, Vol. 64, 1995.
2. Yu Li, "Determination of Asphalt Content and Aggregate Gradation of HMA by Ignition Heating." Thesis for the Degree of Master of Science, Auburn University, AL, March 1992.
3. Nicholas E. Murphy, "Asphalt Content Determination by the Ignition Method." Thesis for the Degree of Master of Science, Auburn University, AL, June 1994.