NCAT Report 95-02

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March 1995

Paper provided to the Association of Asphalt Paving Technologists for presentation and publication. Paper has been tentatively accepted.

National Center for Asphalt Technology
NCAT
AUBURN UNIVERSITY
277 Technology Parkway • Auburn, AL 36830

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Elton R. Brown
Director
National Center for Asphalt Technology
Auburn University, Alabama

Nicholas E. Murphy

Li Yu

Stuart Mager
Graduate Research Assistant
Auburn University, Alabama

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HISTORICAL DEVELOPMENT OF ASPHALT CONTENT DETERMINATION BY THE IGNITION METHOD

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INTRODUCTION

Background

Today, many agencies use chlorinated solvents to physically separate the asphalt cement from the aggregate in hot mixed asphalt (HMA) mixtures. Solvent extraction methods provide two important parameters: asphalt cement (AC) content and aggregate gradation of HMA mixtures. However, the solvents used for extraction tests are expensive, difficult to dispose, and unsafe. Growing health and environmental concerns associated with the use of chlorinated solvents have been major factors driving many agencies to seek alternate methods of AC content determination.

Newer types of solvent, generically called biodegradable solvents, have been used and evaluated by several agencies as a replacement for chlorinated solvents. Biodegradable solvents require a modified extraction procedure for determining AC content and aggregate gradation. The modified procedure is more time consuming and is likely less accurate than the method using chlorinated solvents.

Nuclear asphalt content (NAC) gauges in some places have been replaced by solvent extraction methods to measure AC content. NAC gauges are capable of rapidly measuring the AC content of HMA mixtures with accuracy at least comparable to solvent extraction methods. Although NAC gauges have solved many of the problems associated with solvent extraction methods, these methods do not allow for determination of aggregate gradation. Furthermore, the extraction test is still required for determination of AC content in reclaimed asphalt pavement (RAP).

Due to the fact that NAC gauges and biodegradable solvents have not successfully eliminated the use of chlorinated solvents, other test methods need to be developed and evaluated for AC content determination. One such test is to determine the asphalt content by the ignition method.

Objective

This study was conducted to develop a reliable, detailed test procedure for determining AC content by the ignition method. The goal was to minimize the overall test time as well as technician time and to produce a test method with acceptable accuracy.

Scope

A detailed test procedure for the ignition method was developed based upon previous laboratory studies at NCAT. Using the developed test procedure, an extensive evaluation of the ignition method was performed in the laboratory on mixtures with differing mixture properties (i.e. aggregate type, gradation, and AC content). The purpose of the laboratory investigation was to determine the accuracy of this test method for AC content and gradation determinations. The ignition method, centrifuge extraction method, and NAC gauge were evaluated on five construction projects within the state of Alabama. The test procedure was modified based on this study to improve the accuracy and to reduce test time.

BACKGROUND OF IGNITION METHOD

The ignition method utilizes the mass of the HMA mixture, before and after removal of asphalt cement by burning, as a basis of determining AC content. In 1969, as a continuation of National Cooperative Highway Research Project (NCHRP) 10-4, a study performed by Antrim and Busching at Clemson University suggested the use of the ignition method for determination of AC content (*1*). The NCHRP study revealed that virtually complete combustion of the asphalt cement could be achieved by subjecting the HMA mixture to a high temperature (approximately 843°C (1550°F)) and an excess of oxygen in a special fhmace using butane as a fhel. Although the ignition method as proposed by Antrim and Busching was an innovative method for determining AC content, certain aggregate types were found to contribute substantial errors in AC content determinations.

Antrim and Busching noted that the burning of aggregates at 843°C (1550°F) could possibly result in aggregate mass loss. Although aggregate mass loss of granite gneiss was negligible, limestone lost a significant amount of its oven dry mass afler being heated to 843°C (1550°F) for one hour. Due to the aggregate mass loss of certain aggregate types, the results from ignition testing of 1000 gram HMA samples at 843°C (1550°F) for 30 minutes indicated that the measured AC content could deviate from the true AC content up to one percent.

Based upon a similar concept as the ignition method evaluated by Antrim and Busching, subsequent studies have been conducted at the National Center for Asphalt Technology (NCAT) from 1990 to the present time using a muffle furnace (*2*, *3*). The ignition tests conducted at NCAT have alleviated many of the potential problems identified by Antrim and Busching. Whh the newer apparatus and testing procedure which incorporated a lower temperature setting of 593°C (1100°F), the effect of aggregate type on AC content determination was significantly reduced. Accuracy of AC content determinations and aggregate gradations following ignition of the HMA has been comparable to other test methods.

In the ignition method, the asphalt content is determined from the mass of the HMA sample before and atler burning. The AC content is determined in a way similar to the moisture content of soils. The mass of the HMA mixture and asphalt-free aggregate are used to determine the measured AC content of the mixture by the following equation:

$$P_m = \frac{M_m - M_t}{M_m} * 100 \qquad (1)$$

where,

P_m= the measured AC content (percent) by mass of the mix,
M_m = the mass of the HMA mixture before ignition, and
M_t= the aggregate mass after ignition.

The difference between the mass of the HMA mixture before ignition testing and the aggregate mass after ignition testing is due to removal of asphalt cement and mass loss of aggregate for some aggregate types. As a result of aggregate mass loss, the measured AC content determined by the ignition method has been shown to be slightly greater than the actual AC content for HMA mixtures composed of certain types of aggregates. Correction for the aggregate mass loss for these aggregate types must be determined by burning a mixture with a known AC content (typically without asphalt cement) to determine a correction factor. Further research is being conducted to improve the ignition method so that a correction factor will not be needed.

In 1990-92, NCAT conducted tests on 600 gram HMA samples composed of granite, gravel, and limestone with a muffle firnace at a temperature setting of 593°C (1100°F). It was concluded that the measured AC content for all types of mixtures was within 0.4 percent of actual AC content. The measured asphalt content for all mixtures except those containing limestone were typically within 0.2 percent of the actual AC content.

This early work showed that the ignition method could be used to determine both AC content and aggregate gradation. The method does not require the use of solvents; therefore, no disposal problem is associated with the method.

DEVELOPMENT OF TEST PROCEDURE

Prior to detailed evaluation of the ignition test, it was necessary to develop a test procedure which could be used in the laboratory and field. The test procedure developed for this test would have to incorporate the following:

1. test temperature which would minimize test time while reducing aggregate mass loss;
2. sample size which would provide an adequate amount of aggregate for gradation analysis;
3. apparatus capable of quickly heating large samples to high temperatures; and,
4. method of calibrating for aggregate mass loss (if needed).

Initial Selection of Test Temperature and Test Time

Different types and amounts of asphalt cements were tested under different temperature settings (2). This work showed that small samples of asphalt cement (AC-10, AC-20, and AC-40) could be completely burned at temperatures of 482°C and higher and at test times greater than one hour for the small furnace used at that time.

From the earlier tests at NCAT, the test time decreased greatly when the temperature was increased from 538°C to 593°C; however, the test time only reduced slightly when the temperature was increased above 593°C. For this reason, a temperature setting of 593°C was initially selected as the optimum temperature for the ignition method.

In the early work conducted at NCAT, HMA samples were placed in a shallow porcelain dish for testing. It was noted that the concave shape of the porcelain dish caused the asphalt cement from the HMA sample to flow to the middle and bottom of the container when initially heated. The settlement of the asphalt cement would form a thick layer in the bottom of the dish requiring up to 4.5 hours to remove the asphalt residue to provide asphalt-free aggregates.

Since the porcelain dish created conditions which resulted in increased test time, an effort was made to search for a suitable container. Ezio Santagata conducted limited studies on the ignition method at the Universita' Di Ancona in Italy. He reported that flat, stainless steel pans provided more ideal testing conditions than porcelain dishes (5). Santagata stated that the flat, stainless steel pans allowed more exposure of the asphalt cement to uniform heating conditions thus reducing the required test time.

The largest commercially available size of stainless-steel pans, which would fit into the forced draft furnace, was selected for testing in the 1993 NCAT work (3). For testing of the HMA sample, it was proposed to equally divide the sample into halves and to place each sample half into a separate pan. One pan could be placed on the top shelf and the other on the bottom shelf of the forced-draft furnace. Using two pans would increase the surface area of the asphalt cement and would reduce the testing time.

A preliminary study was performed in the laboratory to evaluate the time required for completing a test under the new testing conditions. The results from the testing indicated that no traces of asphalt cement remained after one hour and forty-five minutes of burning for the samples evaluated. The time needed to complete testing of a 1200 gram sample of HMA was decreased by two hours and forty-five minutes using improved apparatus and methodology. To be conservative, a test time of two hours was selected for both laboratory and field testing.

Additional work at NCAT in 1994 reduced the test time to one hour by using pans with 4.75 mm screen mesh. The screen mesh pans were used with a solid pan underneath to catch any aggregate that fell through the screen mesh. The test time was again reduced to approximately 30 minutes by pulling air through the furnace.

Sample Size

The appropriate sample size required for testing by the ignition method should provide an accurate measure of AC content and an ample amount of aggregate afier testing for gradation analysis. Standard test procedures for gradation analysis in ASTM and AASHTO specify the size or quantity for a sample varying with the nominal maximum-size of the aggregate.

As specified in ASTM C 136 and C 117, the minimum mass requirements of test samples having nominal maximum aggregate sizes of 9.5 mm (3/8 inch)and 19.0 mm (3/4 inch) were 1000 and 2500 grams, respectively (*4*). Most work at NCAT to date has been performed with 1200 gram samples.

Equipment

Studies in 1990-92 at NCAT were performed with a small muffle furnace. Using a temperature setting of 593°C (1100°F), the small muffle furnace was capable of achieving complete combustion of a 600 gram HMA sample in approximately 4.5 hours (*2*). Under these test conditions, it was evident that a larger fimace was needed to reduce testing time.

A forced-draft furnace having sufficient size to test fill size samples was selected for additional testing. The inner chamber dimensions of 35.6 × 30.5 × 33.0 cm (14" × 12" × 13") allowed the testing of 1200 gram HMA samples at 593°C (1100°F). The furnace was programmable to control temperature and heating time to eliminate operator inconveniences and increase reproducibility.

Calibration

A correction factor for aggregate mass loss was included in the test procedure. The correction factor, which is inherently dependent upon aggregate type, accounts for the mass loss in the aggregate at high temperatures. Aggregate mass loss has been shown to increase the measured AC content by up to 0.4 percent above the actual AC content (*2*).

For calibration, a minimum of three samples of known AC content should be prepared, typically with no asphalt cement. The weight loss for the three samples should be determined by the ignition test. The weight loss results should be averaged to determine the correction factor.

IGNITION WORK IN 1993

Laboratory Testing

General Description of Laboratory Testing

A general description of laboratory testing to evaluate the accuracy of ignition method is shown in Figure 1. HMA mixtures containing two different types of aggregate and one type of asphalt cement were prepared for ignition testing to determine AC content and gradation. The materials used to prepare test specimens are described below:

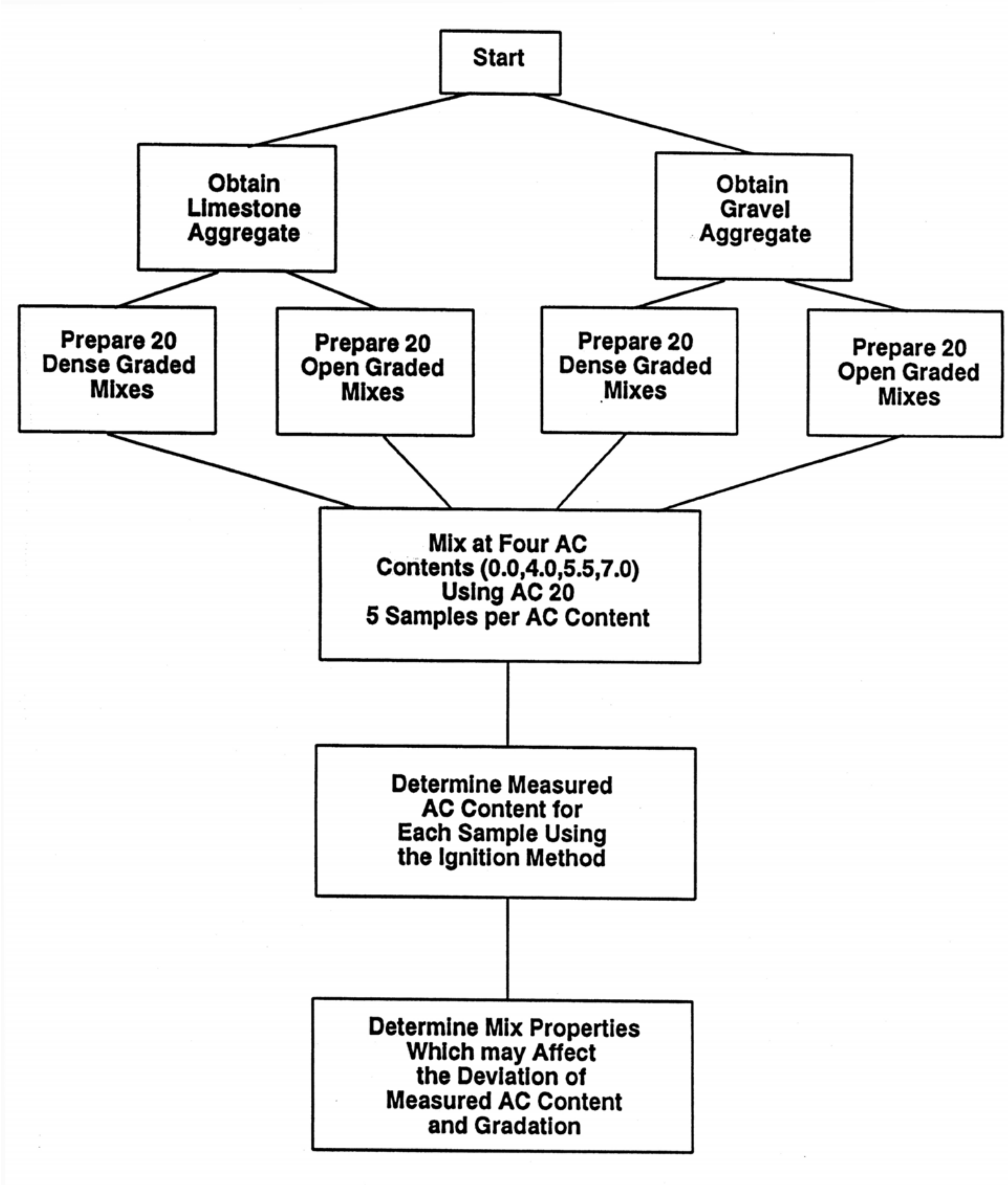

Figure 1. Flowchart of Laboratory Testing

Aggregate Types: Crushed Limestone
Crushed Gravel

AC Grade: Viscosity Graded AC-20

Mixtures were prepared with known AC content and aggregate gradation. Two different gradation types and four different AC contents were used in preparing both limestone and gravel mixtures. Table 1 presents the aggregate type with respective gradation type used to prepare HMA samples at various AC contents.

Table 1. Aggregate Type and Gradation at Various Asphalt Contents

AC Content, percent	Limestone		Gravel	
	Dense Graded	Open Graded	Dense Graded	Open Graded
0.0	5 samples	5 samples	5 samples	5 samples
4.0	5 samples	5 samples	5 samples	5 samples
5.5	5 samples	5 samples	5 samples	5 samples
7.0	5 samples	5 samples	5 samples	5 samples

Aggregate samples were prepared to have a total mass of 1200 grams and to meet the desired gradation. The aggregate gradations used are shown in Table 2.

HMA samples were prepared at four AC contents as shown in Table 1. AC contents ranged from 0.0 to 7.0 percent by mass of the mix. Immediately following the preparation of each sample, the measured AC content was determined by the ignition method.

Table 2. Gradation of Each Aggregate Type

Sieve Size	Dense Graded Limestone	Open Graded Limestone	Dense Graded Gravel	Open Graded Gravel
	Cumulative Percent Passing			
19.0 mm (3/4")	100.0	100.0	100.0	100.0
12.5 mm (1/2")	98.0	95.1	96.5	100.0
9.5 mm (3/8")	88.4	42.7	86.8	94.6
4.75 mm (No. 4)	65.1	22.8	63.4	41.6
2.36 mm (No. 8)	45.4	13.4	44.4	11.4
1.18 mm (No. 16)	31.8	8.5	24.9	10.4
600 microns (No. 30)	25.5	6.4	17.0	9.4
300 microns (No. 50)	20.2	4.9	11.2	6.3
150 microns (No. 100)	13.6	3.0	7.2	3.9
75 microns (No. 200)	8.2	1.8	4.5	2.3

Test Results from Laboratory Testing

Ignition testing was performed on a total of 80 samples to determine the measured AC content of each sample. A washed sieve analysis was performed according to ASTM C 136 and C 117 on the aggregate recovered after burning by the ignition method. A summary of measured AC contents and aggregate gradations is provided in Table 3.

AC Content. The deviation of measured AC content from the true AC content for the 80 samples tested in the laboratory was evaluated to determine the accuracy and precision of the asphalt content by the ignition method. Additionally, the average deviation of measured AC content from the true AC content and the standard deviation of asphalt content were determined for each aggregate type and gradation. These values are presented in Table 3. Using these values, the accuracy and precision of measured AC content determinations by this test method were determined for the various mixtures. The average deviation of measured AC content from the true AC content provides an indication of the accuracy for determining AC content by the ignition method. Although calibration could have been performed on HMA samples tested in the laboratory to increase the accuracy of AC content determination, it was excluded to evaluate the potential for determining AC content by this method without the need for calibration.

The average deviation of measured AC content from the true AC content for the 40 limestone samples was 0.3 percent (Table 3). Based on the test method available in 1993, it appears that a correction factor (involves calibration) would be required for the limestone mixes to account for the average 0.3 percent loss in aggregate mass. This test procedure involved a temperature of 1100°F and a test time of two hours.

Limestone mixtures, as shown in Table 3, have average deviations of measured AC content from actual AC content of approximately 0.37 and 0.24 percent for the dense-graded and open-graded mixtures, respectively. Since the average deviation of measured AC content for the dense-graded limestone mixtures is approximately 0.13 percent greater than that of open-graded mixtures, gradation type may have some effect on the deviation of measured AC content for mixtures composed of limestone. The dense-graded mixture appears to have more aggregate mass loss on ignition.

In contrast to limestone mixtures, the gradation type did not have an appreciable effect on the deviation of measured AC content from the true AC content for gravel mixtures. As shown in Table 3, the average deviations of measured AC content from actual AC content for gravel mixtures were approximately 0.09 and 0.04 for the dense graded and open graded mixtures, respectively. Since the average deviations of measured AC content for gravel mixtures are low and do not vary dramatically between aggregate gradations, the measured AC contents for gravel mixtures accurately depict the true AC content and a calibration to determine a correction factor is not needed. The deviation of measured AC content from the true AC content is generally a function of aggregate mass loss. Aggregate type, gradation type, and AC content may possibly affect the deviation of measured AC content from the true AC content in ignition testing.

Statistical Analysis System (SAS) software was employed to perform the Student Newman Keuls (SNK) method of multiple comparison on the deviation of measured AC content. The deviation of the measured AC content from the true AC content was compared for the four aggregate gradations as shown in Table 4. This comparison was also made for the four AC contents in Table 5.

Table 3. Ignition Test Results from Laboratory Study

Aggregate Type	Grading Type	Number of Samples	Asphalt Content			Percent Passing 75 microns (No. 200)			Percent Passing 4.75 mm (No. 4)		
			Actual	Measured	Stan. Dev.	Actual	Measured	Stan. Dev.	Actual	Measured	Stan. Dev.
Limestone	Dense	5	0.0	0.5	0.04	8.2	7.9	0.3	65.1	64.6	0.3
		5	4.0	4.3	0.05	8.2	8.9	0.4	65.1	64.9	0.3
		5	5.5	5.8	0.06	8.2	9.1	0.1	65.1	64.7	0.1
		5	7.0	7.3	0.00	8.2	9.1	0.2	65.1	64.9	0.2
	Open	5	0.0	0.2	0.00	1.8	1.5	0.2	22.8	23.0	0.2
		5	4.0	4.2	0.04	1.8	3.5	0.1	22.8	23.5	0.7
		5	5.5	5.8	0.09	1.8	3.4	0.3	22.8	23.2	0.4
		5	7.0	7.3	0.06	1.8	3.5	0.1	22.8	23.0	0.2
Gravel	Dense	5	0.0	0.1	0.04	4.5	4.3	0.1	63.4	63.1	0.1
		5	4.0	4.1	0.11	4.5	4.8	0.2	63.4	63.2	0.2
		5	5.5	5.5	0.06	4.5	4.8	0.3	63.4	63.6	0.3
		5	7.0	7.1	0.08	4.5	4.7	0.0	63.4	63.6	0.3
	Open	5	0.0	0.1	0.04	2.3	2.5	0.1	41.6	42.4	0.5
		5	4.0	4.0	0.04	2.3	2.9	0.1	41.6	42.2	0.3
		5	5.5	5.5	0.04	2.3	2.8	0.1	41.6	41.9	0.2
		5	7.0	7.0	0.00	2.3	2.8	0.2	41.6	41.7	0.2

Table 4. Comparison of the Deviation of Measured AC Content From the True AC Content for Various Aggregate Gradations

Test Variable: Deviation of Measured AC Content from the True AC Content			
Aggregate Gradation	**No. of Samples**	**Average Deviation, percent**	**SNK Grouping***
Dense Graded Limestone	20	0.365	A
Open Graded Limestone	20	0.235	B
Dense Graded Gravel	20	0.090	C
Open Graded Gravel	20	0.040	C

* Values of measured AC content deviation with the same letter are not significantly different at the 95 percent level of confidence

Table 5. Comparison of the Deviation of Measured AC Content for Various AC Contents

Test Variable: Deviation of Measured AC Content from the True AC Content			
True AC Content, percent	**No. of Samples**	**Average Deviation, percent**	**SNK Grouping***
0.0	20	0.230	A
4.0	20	0.175	A
5.5	20	0.165	A
7.0	20	0.160	A

* Values of measured AC content deviation with the same letter are not significantly different at the 95 percent level of confidence

Table 4 supports the contention that aggregate gradation of limestone mixtures (the limestone used in this study) can significantly influence the deviation of measured AC content. There was a statistically significant difference between the test results for the dense-graded and open-graded mixtures for the limestone aggregate. There also was a statistically significant difference between the test results for the two aggregate types.

On the contrary, the aggregate gradation of the gravel mixtures did not statistically affect the deviation of measured AC content. The deviation of measured AC content from the true AC content did not differ by more than 0.05 percent between the dense- and open-graded gravel mixtures. Regardless of gradation type, one can assume that AC content determinations can be performed on gravel mixtures (the gravel type used in this study) without calibration.

In the laboratory, each aggregate gradation was prepared using a total of four AC contents (i.e. 0.0, 4.0, 5.5, and 7.0 percent by mass of the mixture). The measured AC content was determined by the ignition method on the HMA samples. The deviation of measured AC content was averaged among the aggregate gradations according to AC content. The SNK method of multiple comparison was performed on the average deviation of measured AC content for various AC contents as shown in Table 5.

The comparison indicates that statistically significant differences do not exist between the deviation of measured AC content from the true AC content for aggregate samples having 0.0 percent AC and those samples having asphalt cement added to aggregate. Increased aggregate mass loss was not recognized for aggregates uniformly coated with asphalt cement as opposed to the aggregate samples. Thus, no obvious relationship exists between AC content and aggregate mass loss of HMA samples. However, the data does show more aggregate weight loss at 0.0% asphalt but the differences are not statistically significant.

The previous study by Antrim and Busching indicated that heating aggregate samples did not subject the aggregate to the same heating conditions which prevailed when burning asphalt mixtures [l]. The results of this laboratory investigation contradicts that of the previous study by Antrim and Busching. A possible cause for this difference is the use of different temperature settings during testing. A temperature setting of 593°C (1100°F) was used in this study while studies by Antrim and Busching were performed at 843°C (1550°F) [l].

Table 3 also shows the standard deviation of the measured asphalt content and measured aggregate gradation. These numbers are all very low indicating that this is a very precise test. A formal round robin study is needed to develop acceptable precision and bias statements.

Aggregate Gradation. As previously discussed, ignition testing at high temperatures may result in aggregate mass loss. Since aggregate mass loss may affect the gradation of the aggregates, an effort was made to examine the gradations of samples following ignition testing. For this study, the values of percent passing the 4.75 mm (No. 4) sieve and the 75 micron (No. 200) sieve were analyzed to determine the effect of the test method on gradation. The analysis of these two critical sieve sizes will provide some indication of the effect on the remaining sieve sizes.

For each of the aggregate gradations in Table 3, the difference between the average percent passing the 4.75 mm (No.4) sieve before and after ignition testing ranges from -0.5 to 0.8 percent. Thus, the percent passing the 4.75 mm (No.4) sieve could be determined with a high degree of accuracy using the ignition method. Table 6 shows that AC content was observed to have no effect on the accuracy of the percent passing the 4.75 mm (No.4) sieve.

The single-operator standard deviation of the percent passing the 4.75 mm (No. 4) sieve was determined to be 0.45 percent for the 80 samples tested in the laboratory. This is very low compared to the typical allowable tolerance for percent passing the No. 4 sieve so the accuracy of the test method appears to be very good.

The precision and accuracy of the percent passing the 75 micron (No. 200) sieve were evaluated. The mix properties which may affect determinations of percent passing the 75 micron (No. 200) sieve are aggregate type and AC content.

For evaluation of accuracy with respect to mix properties, the difference between the average percent passing the 75 micron (No. 200) sieve before and after ignition testing was determined for each AC content and for each aggregate gradation as shown in Table 3. Differences in percent passing the 75 micron (No. 200) sieve were recognized for both aggregate type and AC content.

Table 3 indicates a substantial difference between the average percent passing the 75 micron (No. 200) sieve before and after ignition testing for limestone aggregates mixed with asphalt cement. Typically, the difference in percent passing the 75 micron (No. 200) sieve was 0.8 percent and 1.7 percent respectively for the dense- and open-graded limestone mixes containing asphalt cement. In contrast, differences were not recognized in gravel aggregates mixed with asphalt cement. The difference in percent passing the 75 micron (No. 200) sieve was 0.3 percent and 0.5 percent for gravel aggregates containing asphalt cement.

Table 6. Comparison of the Percent Passing the 4.75 mm (No.4) Sieve for Various AC Contents

Test Variable: Percent Passing the 4.75 mm (No. 4) Sieve				
True AC Content, percent	**No. of Samples**	**Average of Percent Passing 4.75 mm (No. 4) Before Test**	**Average of Percent Passing 4.75 mm (No. 4) After Test**	**SNK Grouping***
0.0	20	48.22	48.26	A
4.0	20	48.22	48.46	A
5.5	20	48.22	48.36	A
7.0	20	48.22	48.28	A

* Values of measured percent passing 4.75 mm (No. 4) with the same letter are not significantly different at the 95 percent level of confidence

The differences in percent passing the75 micron (No. 200) sieve between aggregate types mixed with asphalt cement can be partially explained by the abrasion resistance of each aggregate type. When mixed with asphalt cement, gravel mixtures, which are resistant to abrasion, were not subjected to much aggregate degradation. Unlike gravel, limestone aggregates are not as resistant to abrasion. Thus, limestone aggregates were likely degraded due to abrasion during the mixing operations.

Furthermore, the difference in percent passing the 75 micron (No. 200) sieve of aggregate samples (0.0 percent AC content) and asphalt mixture samples for both limestone and gravel support the abrasion problem. The aggregate samples that were not mixed with asphalt cement were not affected by aggregate degradation due to handling. The difference (actual minus measured after ignition) in percent passing the 75 micron (No. 200) sieve for aggregate samples was very low, approximately 0.3 percent, for both limestone and gravel aggregates.

Overall, the standard deviation of the percent passing the 75 micron (No. 200) sieve was 0.58 for the 80 samples tested in the laboratory. The single-operator standard deviation of the percent passing the 75 micron (No. 200) sieve determined by the ignition method is less than the 0.71 percent required by AASHTO. If the variability in the percent passing the 75 micron (No. 200) for limestone mixtures due to the abrasion during mixing operations were not encountered, the single-operator standard deviation of the percent passing the 75 micron (No. 200) sieve determined by the ignition method would be even less than the measured 0.58 percent. In any event, the ignition method of determining the percent passing the 75 micron (No. 200) sieve attained a high degree of precision.

The SNK method of multiple comparison was performed on the percent of particles passing the 75 micron (No. 200) sieve. The average percent of particles passing the 75 micron (No. 200) sieve size from all aggregate gradations was analyzed for each AC content as shown in Table 7.

The comparison in Table 7 indicates that significant differences exist between the percent passing the 75 micron (No. 200) sieve of those mixtures prepared with 0.0 percent AC and those prepared with other AC contents. As previously explained, the difference between mixes with asphalt cement and mixes without asphalt cement is likely the result of aggregate degradation due to abrasion during mixing operations. If aggregate degradation did not occur during mixing operations, there would likely be no significant differences in the percent of particles passing the 75 micron (No. 200) sieve size between different AC contents.

Table 7. Comparison of the Percent Passing the 75 micron (No. 200) Sieve for Various AC Contents

Test Variable: Percent Passing the 75 micron (No. 200) Sieve				
True AC Content, percent	**No. of Samples**	**Average of Percent Passing 75 micron (No. 200) Before Test**	**Average of Percent Passing 75 micron (No. 200) After Test**	**SNK Grouping***
0.0	20	4.20	4.06	B
4.0	20	4.20	5.02	A
5.5	20	4.20	5.01	A
7.0	20	4.20	5.04	A

* Values of percent passing 75 micron (No. 200) with the same letter are not significantly different at the 95 percent level of confidence

Field Testing

Test Plan for Field Testing

Although the ignition method appeared to be a promising method of AC content determination in the laboratory, research was needed to determine the reliability and reproducibility of the method in a field quality control type of environment. Five ongoing highway projects within the state of Alabama were selected for field testing of the ignition method. Projects were selected on the basis of availability within the East Central region of Alabama.

For each project, approximately 10-12 samples were tested in a period of one week using the NAC gauge, ignition method, and centrifuge extraction. Biodegradable solvents were incorporated as part of NAC gauge testing, since the NAC gauge does not provide asphalt-free aggregate for gradation analysis following AC content determination. Determinations of AC content by the ignition method were performed at the plant along with determinations by the NAC gauge. Centrifuge extractions were performed in the NCAT laboratory on split samples which were representative of those tested at the plant by the NAC gauge and ignition method. By directly comparing the results from the three methods, the ignition method was evaluated for its potential to be used for normal QC/QA of HMA.

General Description of Field Testing

A general description of field testing is shown in Figure 2.

Project Description

A brief description of plant type and mixture type for each project has been provided in Table 8. The description includes facility type, type of mix produced and tested, and the number of samples tested by the NAC gauge, ignition method, and centrifuge extraction.

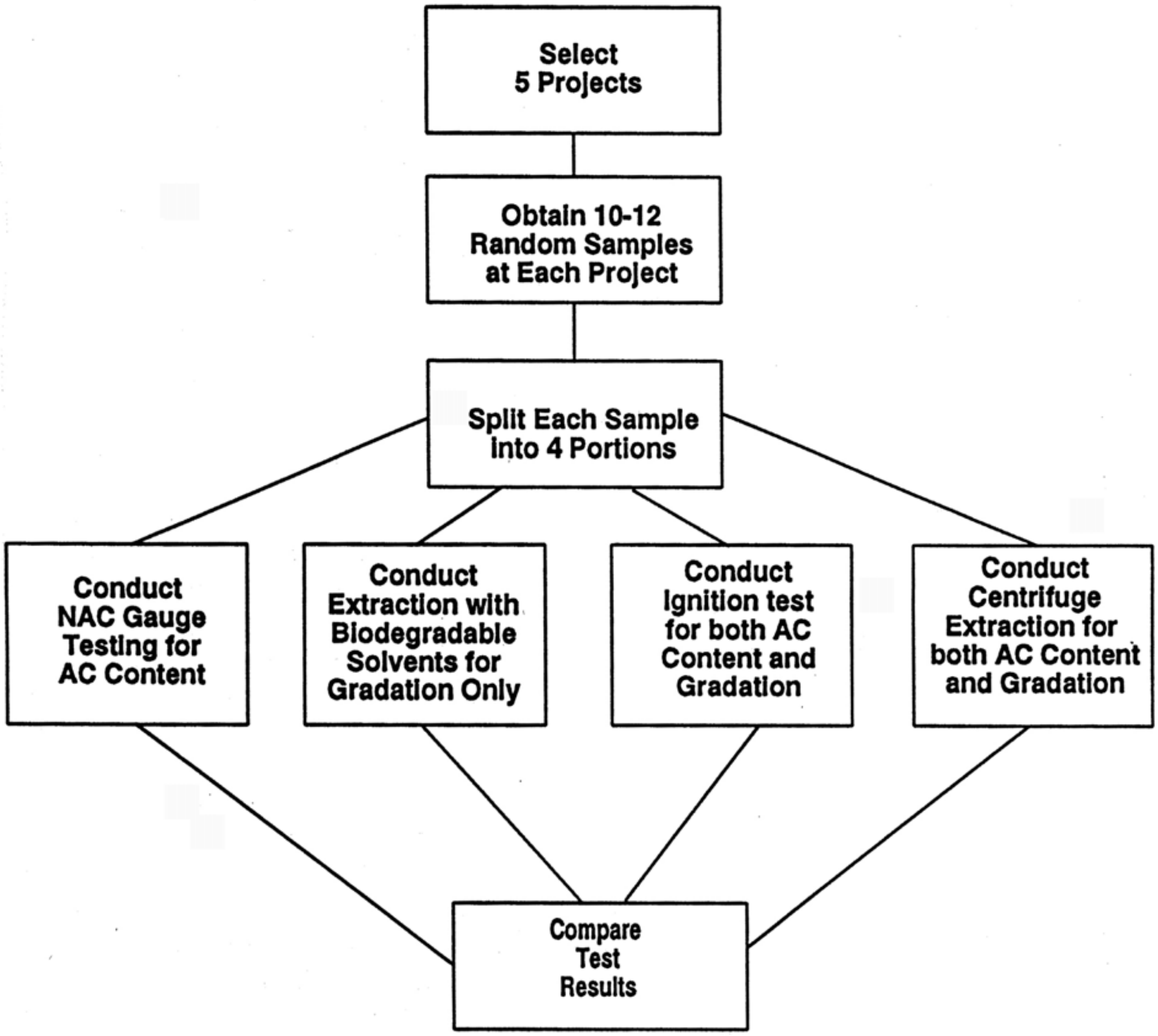

Figure 2. Flowchart of Field Testing

Table 8. Project Description

Project No.	Facility Type	Mix Type	No. of Samples
1	Drum Mix	Surface Course (416 Mix 1)	10
2	Drum Mix	Binder Course (414 Mix 3)	11
3	Batch	Surface Course (416 Mix 4)	12
4	Drum Mix	Surface Course (416 Mix 1)	11
5	Batch	Surface Course (416 Mix 4)	10

Sampling

For each project, approximately 10-12 samples were taken and evaluated in a period of one week. Random sampling times for one day's production were established by an Alabama Department of Transportation (ADOT) official using AHD-210 procedure for selection of random numbers [6]. Complications related to weather, plant breakdowns, paving equipment breakdowns, or other various field problems were encountered on each project which resulted in fewer samples than desired taken during the day. As a result, sampling in addition to those identified by random numbers was required to obtain 10-12 samples in a one week period. Additional sampling times were arbitrarily selected by the contractor laboratory technician at the HMA production facility.

During the specified times, sample units were obtained in accordance with AASHTO T 168 [7]. By a random method, sample units were taken from the truck at the HMA production facility. The sample units were combined to forma field sample whose quantity equaled or exceeded the required amount for testing. The field sample was reduced by quartering to individual sample sizes required for testing in accordance with AHD-l25 [6].

NAC Gauge and Biodegradable Solvent Testing

The NAC gauge was utilized to determine AC content and biodegradable solvents were used to determine aggregate gradation at the HMA facility by a contractor laboratory technician. Both AC content and gradation were determined at the random sampling times as required by the QC/QA program of the ADOT. However, only AC content was determined using the NAC gauge for the additional sampling times. Since biodegradable solvents were not used as part of NAC gauge testing at additional sampling times, aggregate gradation was not determined for these additional samples.

NAC gauge determinations of AC content were performed in accordance with AHD-354 test procedure. When biodegradable solvents were incorporated as part of NAC gauge testing, both AC content and gradation were determined in accordance with AHD-371 [6].

Since the use of biodegradable solvents in conjunction with the NAC gauge is not a currently accepted method for both AC content and gradation determination, furher discussion is warranted for the relatively new test method. In the AHD-371 test procedure for rapid determination of AC content and gradation two representative samples of HMA are secured for testing. A determination of AC content using the NAC gauge is performed in accordance with AHD-354 test method on one of the samples.

Biodegradable solvents were used on the other sample to physically strip the asphalt cement born the aggregate. The HMA sample was placed in a pail and covered with biodegradable solvent. The mixture was then gently agitated frequently with a spatula allowing sufficient time (20-30 minutes for virgin mixtures; 45-60 minutes for recycled mixtures) for the solvents to dissolve the asphalt cement from the aggregate. The extract was decanted by washing with water over a 2.36 mm (No. 8) sieve nested over a 75 micron (No. 200) sieve. Since the minus 75 micron (No. 200) material was lost during the decanting of the extract, the total extracted mass of the aggregate was determined by the following equation [6]:

$$M_1 = M_S\,(1 - AC) \qquad (2)$$

where,
M_l = total extracted mass of mineral aggregate in grams,
M_S = total mass of HMA sample in grams, and
AC = AC content by NAC gauge in decimal form

Using the total aggregate mass (M_l), cumulative masses of asphalt-free aggregate retained on each sieve are converted to percent passing for gradation analysis [6].

Ignition Testing
Using the ignition method, both AC content and gradation determinations were made at the HMA production facility along with NAC gauge determinations of AC content. The procedure for the ignition method given in the Appendix was basically followed for calibration and testing of HMA mixtures. Two exceptions to the method were solid pans were used and the samples were heated in the furnace for two hours. When HMA mixtures contained RAP, the RAP stockpile was sampled and four determinations of the measured AC content were made using the ignition method. For calibration of the ignition method, the contribution of asphalt cement from the IMP to the mix design was established using the average of the four measured AC contents of the RAP samples, rather than the amount of asphalt cement from RAP as specified in the JMFs. The calibration factor for measured AC content correction determined on each project for the ignition method is provided in Table 9. These values were subtracted from the measured AC contents prior to including in the subsequent tables.

The calibration factors for AC determinations, as shown in Table 9, are relatively low values. The calibration factors ranged from -0.1 percent to 0.3 percent for the five projects. Since these values are relatively low, this indicates that the materials used on each of the projects had little effect on AC content determinations by the ignition method.

Table 9. Calibration Factors Determined on Each Project for the Ignition Method

Project No.	Calibration Factor
1	0.2
2	0.1
3	-0.1
4	0.0
5	0.3

Centrifuge Extraction
Centrifuge extractions were performed in the NCAT laboratory on respective samples obtained during field testing of the NAC gauge and ignition method. Extractions were performed in accordance with ASTM D 2172 Method A using 1,1,1-trichloroethane as solvent [4].

Measured Asphalt Content for Projects 1 Through 5
Measured AC contents by the ignition method are typically observed to be slightly higher than those of the NAC gauge (Table 10). Determinations by the ignition method are approximately 0.20 percent more than those of the NAC gauge. It is impossible to determine born this portion of the study which method of measuring asphalt content is more accurate. Further evaluation of the methods in the laboratory are needed to verify which method actually provides the most accurate determination of AC content.

With the exception of Project 3, the results of the ignition method did compare closely with the NAC gauge on every project. However, significant differences were recognized on Projects 2, 3, and 4 between measurements by the ignition method and centrifuge extraction. The differences between measured AC contents for the centrifuge and the ignition methods appear to be related to the incapability of the centrifuge to extract the total amount of asphalt cement from HMA mixtures. Generally, the centrifuge method had more variability than both the NAC gauge and ignition method. AC measurements by the centrifuge method were performed in the NCAT laboratory by more than one technician which may have increased the variability of the

Table 10. Comparison of Measured AC Contents for the Five Field Projects

Project	Method	No. of Samples	Job Mix AC Content, percent	Average Measured AC Content, percent	Standard Deviation	SNK Grouping*
1	NAC Gauge	10	6.20	6.23	0.183	A
	Ignition	10	6.20	6.44	0.117	A
	Centrifuge	10	6.20	6.50	0.424	A
2	NAC Gauge	11	4.10	4.24	0.246	A B
	Ignition	11	4.10	4.44	0.266	A
	Centrifuge	11	4.10	4.15	0.211	B
3	NAC Gauge	12	5.35	5.32	0.127	B
	Ignition	12	5.35	5.87	0.337	A
	Centrifuge	12	5.35	5.37	0.452	B
4	NAC Gauge	11	5.70	5.81	0.122	A
	Ignition	11	5.70	5.79	0.270	A
	Centrifuge	11	5.70	5.38	0.306	B
5	NAC Gauge	10	5.45	5.45	0.255	A
	Ignition	10	5.45	5.60	0.442	A
	Centrifuge	10	5.45	5.39	0.672	A

* Measured AC contents with the same letter within each project are not significantly different at the 95 percent level of confidence

measured AC content by the centrifuge extraction method. Since there is no way to determine the true variability of the mixture, one cannot say that the method with the lowest variability is necessarily the best method.

The SNK method of multiple comparison, as shown in Table 11, was used to evaluate the measured AC contents by the NAC gauge, ignition method, and centrifuge extraction among all five projects. The results of the multiple comparison indicate that no significant differences exist between the three methods of AC content determination at the 95 percent level of confidence. If these five projects are assumed to be representative of those encountered in the field, determinations of AC content by the ignition method can be considered comparable to those determinations by the NAC gauge or centrifuge extraction.

Measured Gradation for Projects 1 Through 5

Gradation determinations were performed on each sample tested by both the ignition and centrifuge extraction methods. However, aggregate gradations were not determined with the biodegradable solvent method each time a NAC gauge test was conducted. As a result, gradation determinations cannot be directly compared for every sample on each project. Despite the lack of gradation determinations along with NAC gauge testing, gradations from biodegradable solvent, ignition and centrifuge extractions can be compared for those samples taken during QC/QA testing. For those samples taken during QC/QA testing, the percent passing the 4.75 mm (No. 4) and 75 micron (No. 200) sieve sizes are suggested for comparison purposes between the biodegradable solvent, ignition, and centrifuge extraction methods. The percent passing these sieve sizes for each sample is an indicator of the percent passing other sieve sizes.

Table 12 was developed from determinations of percent passing the 4.75 mm (No. 4) sieve by biodegradable solvent, ignition, and centrifuge testing. The SNK method of multiple comparison was performed for determinations of percent passing the 4.75 mm (No. 4) sieve to indicate if methods of determination statistically differed on each project. Determinations of percent passing the 4.75 mm (No. 4) sieve were also compared statistically among all five projects in Table 13.

Table 11. Comparison of Measured AC Contents Among Projects 1 Through 5

Projects 1 Through 5 - Test Variable: Measured AC Content				
Method	**No. of Samples**	**Average Job Mix AC Content, percent**	**Average Measured AC Content, percent**	**SNK Grouping***
NAC Gauge	54	5.36	5.39	A
Ignition	54	5.36	5.62	A
Centrifuge	54	5.36	5.34	A

* Measured AC contents with the same letter are not significantly different at the 95 percent level of confidence

Table 12. Comparison of the Percent Passing the 4.75 mm (No. 4) Sieve for Projects 1 Through 5

Project	Method	No. of Samples	Job Mix AC Content, percent	Average Measured AC Content, percent	Standard Deviation	SNK Grouping*
1	Biodegradable	5	81.0	83.4	3.507	A
	Ignition	5	81.0	86.6	0.548	A
	Centrifuge	5	81.0	85.2	2.387	A
2	Biodegradable	6	48.0	47.2	3.312	A
	Ignition	6	48.0	48.2	4.622	A
	Centrifuge	6	48.0	48.0	3.521	A
3	Biodegradable	9	67.0	67.8	2.538	B
	Ignition	9	67.0	72.7	1.936	A
	Centrifuge	9	67.0	72.0	2.598	A
4	Biodegradable	5	63.0	59.8	3.271	A
	Ignition	5	63.0	63.2	4.438	A
	Centrifuge	5	63.0	60.0	2.236	A
5	Biodegradable	6	63.0	66.0	3.521	A
	Ignition	6	63.0	66.7	3.445	A
	Centrifuge	6	63.0	68.2	3.710	A

* Values of percent passing 4.75 mm (No. 4) with the same letter are not significantly different at the 95 percent level of confidence

Table 13. Comparison of the Percent Passing the 4.75 mm (No. 4) Sieve Among Projects 1 Through 5

Projects 1 Through 5 - Test Variable: Percent Passing 4.75 mm (No. 4)				
Method	**No. of Samples**	**Average Job Mix Percent Passing 4.75 mm (No. 4)**	**Average Measured Percent Passing 4.75 mm (No. 4)**	**SNK Grouping***
Biodegradable	31	64.4	64.7	B
Ignition	31	64.4	67.5	A
Centrifuge	31	64.4	66.8	A

* Values of percent passing 4.75 mm (No. 4) with the same letter are not significantly different at the 95 percent level of confidence

The determinations of percent passing the 4.75 mm (No. 4) sieve by the ignition method compared closely to those of the other methods. The ignition method consistently followed the trend of those determinations by biodegradable solvents and centrifuge extractions. However, differences were realized between determinations by the three methods on some projects.

On Project 3, determinations of the measured percent passing the 4.75 mm (No. 4) sieve by biodegradable solvents were on the average five percent less than those determined by both the ignition method and centrifuge. Analysis of the data shows that the gradations determined by the biodegradable solvents were significantly lower than that measured with the other two methods for Project 3. Close agreement of the standard deviation was attained by each method on the five projects except for Projects 1 and 4.

In addition to the comparison of methods on an individual project basis, the percent passing the 4.75 mm (No. 4) sieve as determined by each method was compared among the five projects as shown in Table 13. The SNK method of multiple comparison indicated biodegradable solvent determinations were significantly lower than those of the ignition method and centrifuge extraction at a 95 percent confidence level. Even though the tests using the biodegradable solvents were lower than the other two methods these results were actually closer to the JMF.

Table 14 provides a summary of all the data on percent passing the 75 micron (No. 200) sieve as determined by biodegradable solvent, ignition, and centrifuge testing. The methods of gradation determination were evaluated statistically on each project using the SNK method of multiple comparison. Additionally, determinations of percent passing the 75 micron (No. 200) sieve by the three methods were statistically compared among all five projects in Table 15.

Multiple comparisons of Project 2 indicated that determinations by the centrifuge method were significantly higher than both biodegradable solvent and ignition method determinations. On project 4, biodegradable solvent determinations were significantly lower than both ignition method and centrifuge extraction determinations.

The SNK method of multiple comptuisons was used to evaluate the determinations of percent passing the 75 micron (No. 200) sieve among the five projects as shown in Table 15.

Table 14. Comparison of the Percent Passing the 75 micron (No. 200) Sieve for Projects 1 Through 5

Project	Method	No. of Samples	Job Mix Percent Passing 75 micron (No. 200)	Average Measured Percent Passing 75 micron (No. 200)	Standard Deviation	SNK Grouping*
1	Biodegradable	5	7.20	8.23	0.551	A
	Ignition	5	7.20	8.68	0.449	A
	Centrifuge	5	7.20	8.62	0.311	A
2	Biodegradable	6	4.50	4.80	0.469	B
	Ignition	6	4.50	4.85	0.399	B
	Centrifuge	6	4.50	5.40	0.283	A
3	Biodegradable	9	5.40	5.03	0.312	A
	Ignition	9	5.40	5.42	0.504	A
	Centrifuge	9	5.40	5.33	0.374	A
4	Biodegradable	5	6.50	6.12	0.363	B
	Ignition	5	6.50	7.10	0.561	A
	Centrifuge	5	6.50	6.94	0.550	A
5	Biodegradable	6	5.80	6.07	0.599	A
	Ignition	6	5.80	5.75	0.485	A
	Centrifuge	6	5.80	5.88	0.479	A

* Values of percent passing 75 micron (No. 200) with the same letter are not significantly different at the 95 percent level of confidence

Table 15. Comparison of the Percent Passing the 75 micron (No. 200) Sieve Among Projects 1 Through 5

Projects 1 Through 5 - Test Variable: Percent Passing 75 micron (No. 200)				
Method	**No. of Samples**	**Average Job Mix Percent Passing 75 micron (No. 200)**	**Average Measured Percent Passing 75 micron (No. 200)**	**SNK Grouping***
Biodegradable	31	5.88	5.73	A
Ignition	31	5.88	6.17	A
Centrifuge	31	5.88	6.24	A

* Values of percent passing 75 micron (No. 200) with the same letter are not significantly different at the 95 percent level of confidence

Analysis of the five projects indicates no significant difference of percent passing the 75 micron (No. 200) sieve for biodegradable solvents, ignition method, or centrifuge extraction.

It is impossible to select the best method to determine asphalt content and gradation from these five field projects. The data does show, however, that the ignition method compares favorably with the other two methods evaluated.

WORK ON IGNITION IN 1994

After work on ignition during 1990-1993 there were several concerns involving the test that needed to be addressed. The earlier work showed that this test method could be used to determine aggregate gradation and asphalt content but some problems needed to be solved before it would be adapted for use.

At the end of 1993 the test time required was too long (2 hours). This had to be shortened. During the burning process, smoke was produced and a method to remove or prevent smoking was needed. It appeared that a calibration would be required for the asphalt content but maybe not for the aggregate gradation. The sample size that had been evaluated was 1200 grams and this needed to be increased for some mixes up to approximately 2500 grams.

The goal was to reduce the test time to approximately 30 minutes or less. The first step in reducing the test time was to use pans made of wire mesh. This allows air to flow freely through the pans exposing more asphalt cement surface area and reducing the ignition time. The procedure involves using 2 pans, one on top of the other, with approximately 1/2 of the sample in the top pan and 1/2 in the lower pan. A 50 mesh screen was originally used for the pans. This approach reduced the required test time from two hours to less than one hour. To insure complete burning, testing is normally conducted for one hour. After conducting several tests with the 50 mesh pans, it was noticed that the screens began to plug with material that could not be removed. Heating the aggregates to this high temperature resulted in some bonding of the fine aggregate to the fine wire mesh. The screen mesh in the pan was then changed to 4.75 mm openings and this problem was solved. Since there is a solid pan underneath the two wire mesh pans, any material that falls through the screens is caught in the solid pan.

Another attempt that has been made to reduce test time has been air injection inside the furnace: A furnace being used by NCAT has variable flow air injection. This same furnace also has scales underneath the furnace that can weigh the sample during the ignition test. Figure 3 shows two tests that were conducted on identical samples of asphalt mixtures at 5 liters per minute (lpm) and 15 lpm air flow rates. These two tests were conducted at 1000°F on mixtures having 6.0 percent asphalt and limestone aggregates. The tests showed that the test conducted at 5 lpm took approximately one hour to remove the 6.0 percent asphalt and the test conducted at 15 lpm took

40-45 minutes to remove the 6.0 percent asphalt. This indicates that air flow has the potential to reduce the test time but one must be careful to ensure that the air flow rate is not so high to cause loss of fines from the mixture. It is also possible that some air flow may result in more complete combustion and thus reduce the smoke that has to be removed. Figure 3 also indicates that most of the asphalt cement is removed during the first 12 minutes or so of the test. This is the period of time when the asphalt cement ignites and a majority of the material is burned from the mixture. It is assumed that the asphalt cement that is not on the surface of the mixture is removed at a slower rate after the first 12 minutes. Both samples had identical weight losses up to 12 minutes, then the mix being tested at an air flow rate of 15 lpm continued to lose asphalt cement at a higher rate. If some mixing of the sample can be performed at 12 minutes this would likely expedite the test.

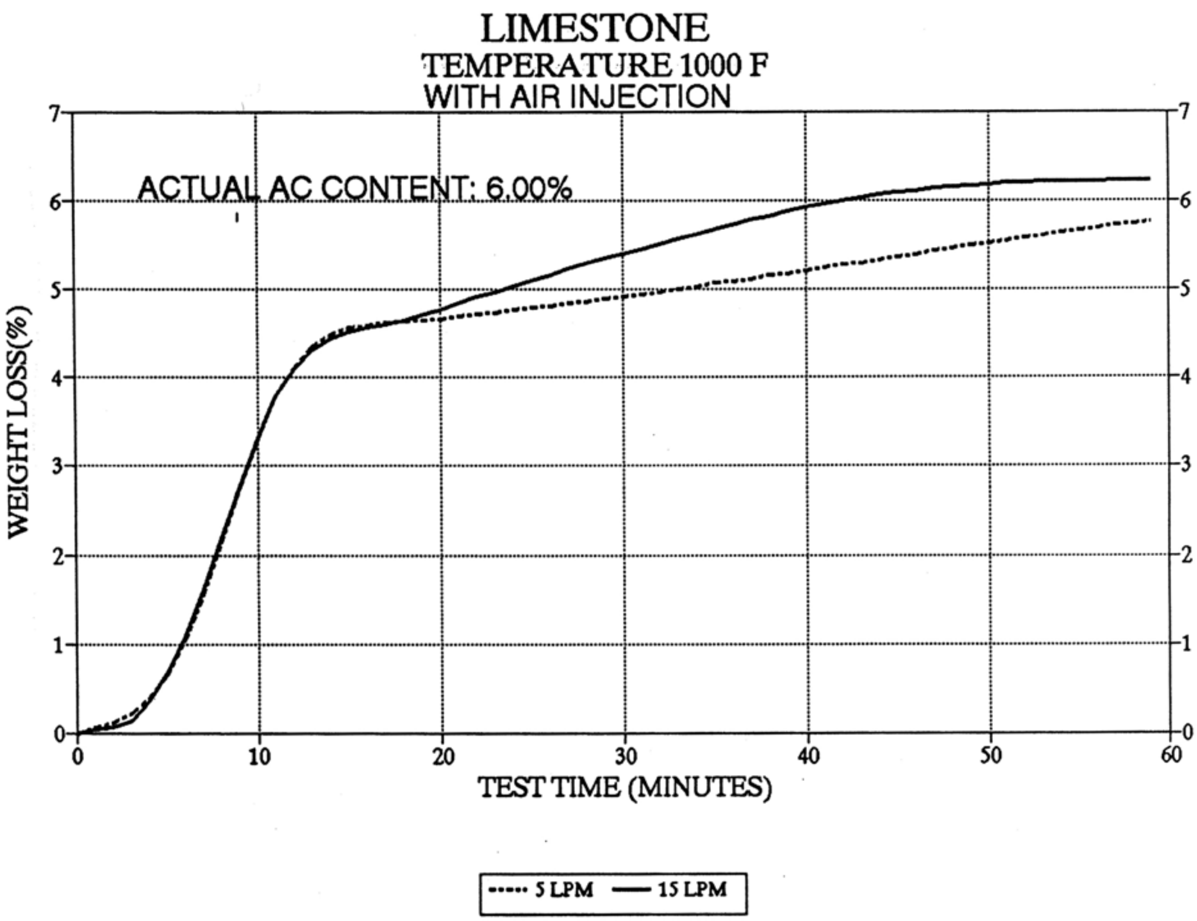

Figure 3. Effect of Air Flow Rate on Removal of Asphalt Cement by Ignition

A method is needed to remove the smoke during the test procedure. NCAT conducts all tests in the laboratory under a hood, but a method is needed for smoke removal at a field laboratory. The latest piece of equipment, that has been used, has a filter that is estimated to remove 90% of all smoke. This equipment pulls the air through the furnace which results in less escape of fumes into the room during the test. With this latest equipment, a 1200 gram sample can be tested in approximately thirty minutes and a 2400 gram sample can be tested in approximately forty-five minutes.

Test data indicates that the measured loss of asphalt cement on heating is very consistent for a given mixture. However, data has indicated that some aggregates tend to lose weight on heating to the high temperatures and thus a correction factor must be made to the measured asphalt content. This can be handled by calibrating for the mixture being evaluated but it is preferred that a test method be developed that needs no calibration. Reducing the test time from two hours to one hour has reduced the amount of calibration necessmy but has not solved the problem for some aggregates. Figure 4 shows the effect of test time and temperature on the weight loss of the HMA mixture. Each point is the average of three tests. This indicates that for the limestone mix evaluated, a test temperature of 1000°F is sufficient to remove the asphalt cement in 45 minutes to one hour without loss of aggregate. For this limestone mix the test can be conducted at 1000°F without the need for a calibration. For some other aggregates the temperature may have to be lowered to prevent the need for a calibration.

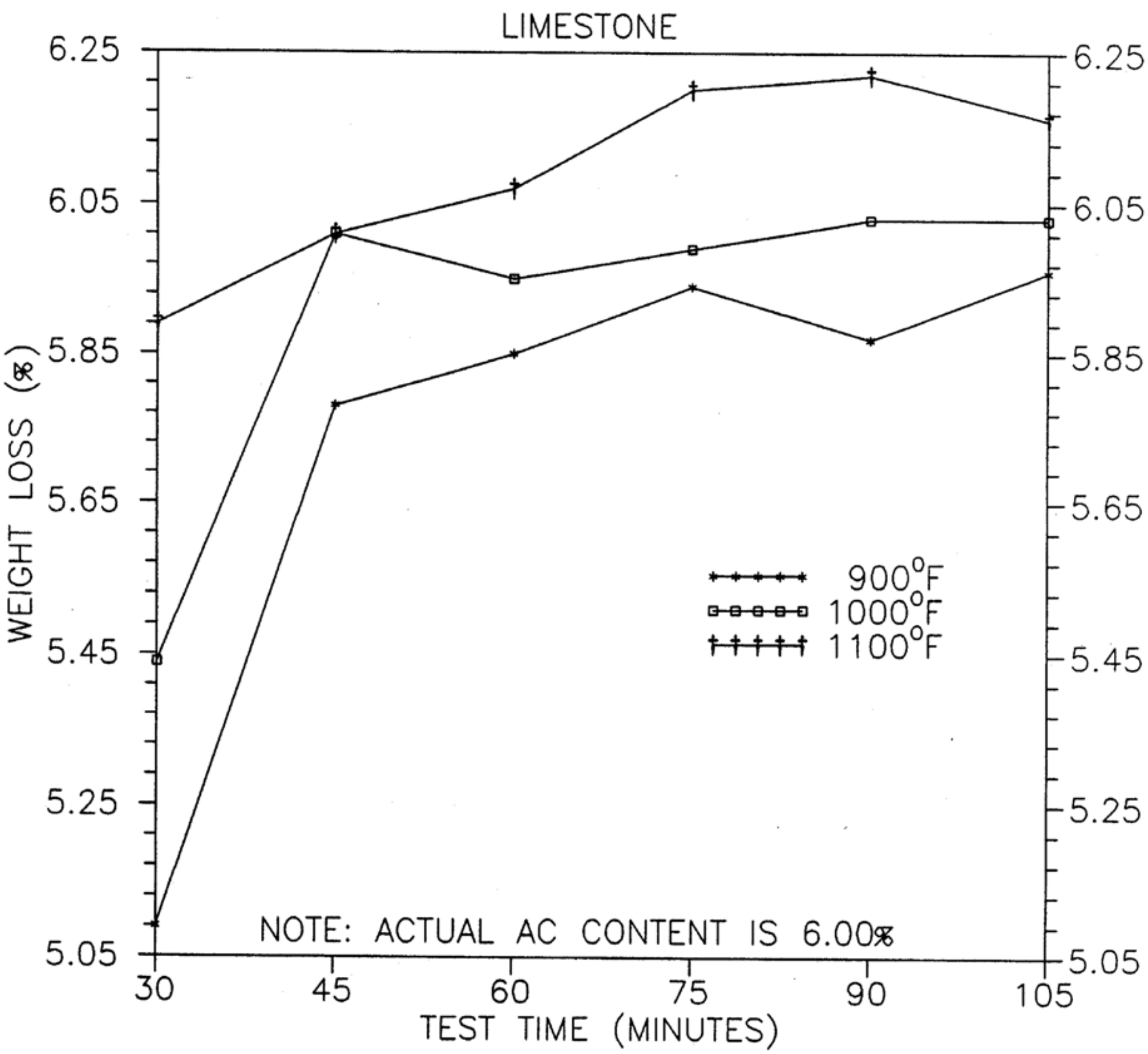

Figure 4. Effect of Test Time and Temperature of Weight Loss

A piece of equipment (consists of furnace, scales, computer, filter) has been produced by an equipment company that will measure and record the asphalt content in approximately 30 minutes. This equipment will also remove approximately 90% of the generated smoke. Twelve machines have been purchased by NCAT for a round robin series that should be completed in early 1995.

CONCLUSIONS AND RECOMMENDATIONS

General

This study examined the use of the ignition method for determining AC content and aggregate gradation. Based on this laboratory and field work, the following conclusions and recommendations are made.

Conclusions

1. The ignition method can be successfully used to determine the AC content and aggregate gradation of HMA mixtures. For some aggregates a calibration is needed to accurately determine the asphalt content. Test results indicate that no calibration is necessary for aggregate gradation.
2. Laboratory work indicates that the accuracy of this test is at least as good as current extraction methods. No solvents are required; thus, this method solves an existing waste disposal problem.
3. The test procedure for the ignition method is relatively simple. The test can be conducted in approximately 30 minutes for asphalt content and requires very little technician time.
4. Work in the field showed no apparent problems with the test method.

Recommendations

1. A testing machine has been developed as a result of this study. A round robin series is needed to verify the accuracy and precision of the test. This study is planned and should be completed in early 1995.

ACKNOWLEDGMENTS

The authors thank the NAPA Research and Education Foundation and the Alabama Department of Transportation for their financial support in this contract. The authors also thank the five contractors in Alabama that allowed NCAT to test mixtures on their construction projects and to compare these test results with their quality control data.

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