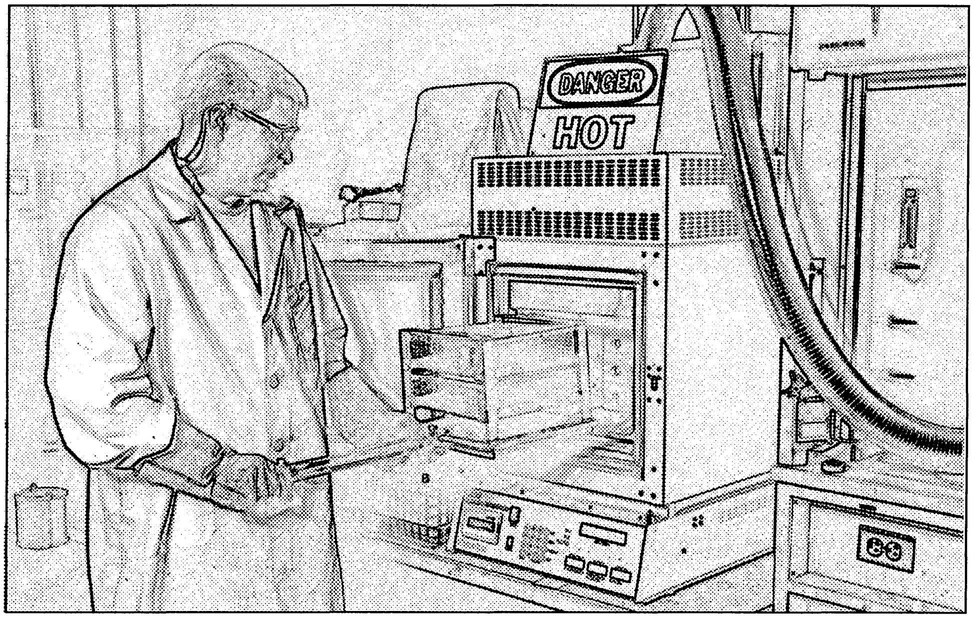
FINAL REPORT

ALTERNATE METHODS OF ASPHALT CONTENT DETERMINATION



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16. Abstract

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Four aggregate types were evaluated using typical surface and base mix gradations found in Virginia. Samples for evaluation were produced at four asphalt contents for each mix design representing typical field variation around the optimum. Operator variance samples were tested non-destructively in the nuclear gauge, and then destructively tested by solvent extraction and the ignition method. In the field validation phase, four methods were used for testing. The gradations of these samples are compared.

As a result of the research, a test method was developed for the ignition furnace with an internal weighing system. This method has been specified as a replacement for solvent extraction in Virginia.

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Virginia Transportation Research Council
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Virginia Department of Transportation and
the University of Virginia)

In Cooperation with the U.S. Department of Transportation
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ABSTRACT

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ALTERNATE METHODS FOR ASPHALT CONTENT DETERMINATION

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INTRODUCTION

Hot-mix asphalt is used to surface 96% of all paved roads in the United States (NCAT, 1991). The asphalt content of these mixtures is critical to their performance, affecting the pavement's tendency for permanent deformation, fatigue life, and susceptibility to moisture damage.

Trichloroethane, a solvent, has been used for years by the Virginia Department of Transportation (VDOT) to extract bituminous binders from hot-mix asphalt (HMA) samples according to VTM-36 (AASHTO T-170). Such samples are used to determine the asphalt content of the mix, and allow aggregate gradations to be run on the extracted mix. The results of these tests influence contractor payment (VDOT, 1991).

The Environmental Protection Agency's (EPA) final ruling on Section 606 of the 1990 Clean Air Act Amendments mandated that the production of Methyl Chloroform (better known as trichloroethane) be discontinued after December 31, 1995 (EPA, 1993). This legislation was in response to the United States' obligation to the fourth meeting of the Montreal Protocol in Copenhagen, November 1992. Article Six of the Montreal Protocol required parties to the protocol to continue to meet and determine adjustments to the production and consumption of ozone depleting compounds (United Nations Environmental Programme, 1987). Under the EPA's ruling on Section 606, Methyl Chloroform was added as a Class I substance on the list of controlled substances.

VDOT examined four methods to replace solvent extractions. Two of these methods, nuclear asphalt content measurement using Marshall sample plugs and the ignition method, were the focus of this study. The other two methods, nuclear asphalt content measurement using sample pans and vacuum extraction with biodegradable solvents, are summarized below.

Nuclear Asphalt Content Measurement Using Sample Pans

Nuclear asphalt content gauges operate on the principal of neutron moderation, where high energy neutrons are thermalized or slowed by atomic collision. The number of collisions required for thermalization decreases with decreasing atomic weight. The thermalized neutrons can be detected by a Helium-3 detector and registered as a count. Prior to measuring the asphalt content of a sample, a calibration curve must be determined from a regression analysis of the registered counts versus asphalt content. Because of the time involved in preparing and testing calibration specimens, a procedure of calibration transfer was developed for transferring a calibration from one gauge to another. Calibration transfer involves determining a relational curve between two different gauges by taking readings on a minimum of six samples of varying asphalt contents in each gauge.

This technique was used by the VTRC in the 1991 and 1992 pilot studies (Hughes, 1991, 1994), allowing the contractor to prepare and run calibration samples in their gauge and use the relational curve to transfer that calibration to VDOT's gauge. An analysis of the pooled standard deviations was conducted on the 1992 data and is presented below as a measure of the typical variabilities that could be used in a Percent Within Limits specification. The data was combined for surface (SM) and intermediate (IM) mixes with 12.5 mm (½ in) and 19.0 mm (3/4 in) nominal maximum size aggregates respectively, based on 172 total samples from 7 contractors. The data for base mixes (BM) with a nominal maximum size of 25.4 mm (1.0 in) is reported separately.

Table 1
Asphalt Content: Pooled Standard Deviation, % 1992 Pilot Study (Hughes, 1994)

Mix Type	Nuclear		Reflux	
	VDOT	Contractors	VDOT	Contractors
SM & IM	.21	.23	.23	.16
BM	.29	.34	.31	.25

Though the nuclear gauge with sample pans appears to provide similar variability to reflux extraction, three problems persist: sample size, lack of gradation, and operator-induced malfunction. For a surface mix, VDOT's volumetric specification, based on the percent within limits concepts outlined in the 1994 AASHTO QA Guide specifications, requires that each time a sub-lot is tested, three 16 kg (35 lb) samples be taken, one for production testing by the contractor, one for possible monitor testing by VDOT, and one to be retained for possible referee testing. Almost half of this sample is to accommodate the +7,000 g (15.4 lb) required for nuclear asphalt contents with the sample pans. The sample size increases to 27 kg (60 lbs) to accommodate base mixes. Secondly, though gradation is de-emphasized under the volumetric specification, it is still useful for plant quality control (QC) and equipment would still have to be available to perform it. Finally, operator-induced errors using cross-calibration were observed in the 1991 pilot study and in subsequent uses of the gauge. The first problem, sample size, could be eliminated by using the sample plug fixture available with Troxler's 3241-C gauge with the Universal Sampling System. The third problem, operator error, could be overcome with training.

Vacuum Extraction with Biodegradable Solvents

Several biodegradable solvents were used by the Materials Division with a vacuum extractor for asphalt content determination. Though this method may provide an accurate asphalt content and allow a gradation to be determined, it has several drawbacks. The method is time consuming. The extraction portion typically takes between 1 ½ and 2 ½ hours. During the first 45 minutes the sample is merely left to soak in the solvent, but the remaining time requires constant attention by a technician, stirring the sample and the filter media to prevent clogging. Following extraction, the aggregate and filter media are dried for a further 1 ½ hours. Thus it takes between 3-4 hours to determine the asphalt content. In some cases the extracted aggregate was placed in a muffle furnace at 427 °C (800 °F) for an additional hour to burn off any residual asphalt prior to a gradation analysis. The method is also expensive. The test costs upwards of $30.00 per sample, including the filter media, solvent, and the disposal of the residual solvent, asphalt and water mixture.

PURPOSE AND SCOPE

The purpose of this study was to evaluate potential methods of asphalt content determination and recommend a method to replace solvent extraction for use in Virginia. Since sufficient work had already been completed on the nuclear method with sample pans, the study initially focused on the nuclear method with Marshall plugs. The goal was to optimize the test method, including the calibration procedure; examine the effects of various aggregates on the accuracy of the gauge with sample plugs; and conduct a round-robin to determine the testing variance. Concurrently, the VDOT Materials Division examined vacuum extraction and the National Center for Asphalt Technology (NCAT) developed the ignition method. Work parallel to NCAT's was conducted at VTRC using four different furnaces. Based on the results of the early work with the furnace, including field trials, and the NCAT round-robin with the Thermolyne ignition furnace, a test method was developed. The ignition method was accepted as an alternate method of asphalt content determination for the 1995 construction season.

METHODOLOGY

General

Four coarse aggregate types from Virginia, with a range of absorptions, were sampled. Table 2 presents the types, bulk specific gravities and absorptions. Seven contractors' mix designs were used to blend the aggregates. The mix designs corresponded to three standard VDOT mixes (VDOT, 1991): surface mix (SM) -2C, base mix (BM) - 2, and BM-3. SM-2C is a 75-blow Marshall designed mix used primarily on interstate and primary routes. BM-2 and BM-3 have a minimum asphalt content (by weight of mix) of 4.4% and 4.0% respectively. The gradation ranges for all three mixes are presented in Table 3. Each aggregate type was used in both a surface and base mix, with the exception of the Bluefield limestone.

Enough aggregate was obtained to prepare all the specimens from aggregate sampled at a single date. The individual aggregates were blended according to the mix design proportions and sieved into individual size fractions. The size fractions were recombined to meet the contractors mix design. All samples were batched and mixed on an individual basis. An extra specimen or

Table 2.
Coarse Aggregate Properties

Aggregate Location and Type	Bulk Specific Gravity	Absorption, %
Shadwell Greenstone	2.96	0.5
Grottoes River Gravel	2.56	1.8
Boscobel Granite/Gneiss	2.61	0.7
Bluefield Limestone[1]	2.71	0.3

[1] Only used in base mixes

Table 3.
Gradation Design Range

Sieve Size	SM-2C Percent Passing	BM-2 Percent Passing	BM-3 Percent Passing
51 mm			100
37.5 mm		100	97-100
25 mm		97-100	70-86
19 mm	100	75-90	
12.5 mm	97-100		46-64
9.5 mm	82-94	54-74	
4.75 mm	48-62		26-44
2.36 mm		30-38	
0.6 mm	18-24		8-18
0.075 mm	4-7	3-6	2-5

"butter" mix was prepared before mixing any of the test specimens. The butter mix was used to coat the mixing bowl with a thin coating of asphalt and fines that would typically be left after mixing a specimen. Sample plugs used for nuclear gauge testing were compacted according to AASHTO T 245. All mix designs were confirmed before any asphalt content testing. The SM-2C samples were mixed with AC-30. The BM-2 and BM-3 samples were mixed with AC-20.

Nuclear Asphalt Content Measurements

The Troxler Model 3241-C gauge with the Universal Sample System™ evaluated in this paper meets all of the requirements designated by ASTM 4125-94, *Standard Test Method for Asphalt Content of Bituminous Mixtures by the Nuclear Method* (ASTM, 1994). The gauge has two unique features which were evaluated in this study: the ability to test 100 and 150 mm Marshall plugs, and the ability to determine a calibration transfer between two different gauges.

Three point calibrations were run with samples prepared at ±1.0% and at the optimum asphalt content. Three Marshall plugs were prepared for each asphalt content of the surface mix calibrations. The two plugs closest in weight to each other and closest in weight to the average of all of the pairs of calibration specimens were used. The calibration was performed on the morning after the samples were compacted. After compaction the samples were stored in front of a fan until testing. The same schedule of compaction, cooling, and testing was used for the remaining experiments. A four minute count period was used for the calibrations.

The gauge is designed to take readings with sample plugs in either 1, 4, or 8 minute intervals. Four measurement intervals make up one test. At the end of the first interval the sample is rotated 180°. At the end of the second the sample is turned over. At the end of the third interval the sample is again rotated 180°. The total test time ranges from 4 to 32 minutes, not including the time needed to manipulate the samples. In the first experiment, an attempt was made to optimize the test time. Seven samples of varying asphalt content were prepared with Shadwell Greenstone conforming to both SM-2C and BM-2 gradations, with 100 mm specimens prepared for the SM-2C and 150 mm specimens prepared for the BM-2. Each sample was tested three times by the same operator.

There has been debate in the literature as to whether variances in the weight or the volume of the sample affect the accuracy of the readings. Though ASTM 4125-94 specifies that the test samples must be ±10 g of the calibration specimens, there is no documented research to support these numbers. The second experiment sought to determine an acceptable range of variation in weight which would not degrade the sample accuracy. Pairs of surface mix samples were prepared at ±1, ±10, and ±25 g of the pair of calibration specimens at the design asphalt content. Each pair of surface mix samples was tested 5 times. The order of testing (by weight) was randomly determined for each replicate set. Samples of both SM-2C and BM-2 were prepared using Shadwell Greenstone. All of the samples were mixed, compacted and tested by one operator.

The third experiment evaluated operator variance, effects of aggregate type, and variability over a range of production asphalt contents. Four aggregates described above were used with three SM-2C mix designs, 3 BM-2 mix designs, and 1 BM-3 mix design. Three operators mixed, compacted and tested specimens at ±0.2% and ±0.5% of optimum asphalt content. Each set of specimens was tested three times (non-destructive replicates). The order of testing was randomly determined within each replicate set.

Asphalt Content by the Ignition Method

Four furnaces were used throughout the study. The first two were muffle ovens with small chamber sizes which operate in a limited oxygen or reducing atmosphere (a second muffle furnace was purchased when the temperature controller failed on the first and burnt out all of the heating elements). Experimentation with the muffle furnaces grew out of Florida DOT's use of them for burning off residual asphalt remaining after vacuum extraction and NCAT's study of the ignition method. One operator's samples from the nuclear gauge operator variance experiment were heated at 140 °C for 45 minutes to drive off any moisture and soften the compacted samples. The two SM samples or single BM sample were mixed and quartered. Opposite quarters were tested using reflux extraction (ASTM D2172) and burnt in the muffle furnace. The muffle furnace tests were conducted at 427 °C in Pyrex evaporation dishes 22 cm in diameter. It was difficult to remove all of the asphalt without stirring the sample. Also, the chamber blower was not designed to handle the ignited asphalt residues and quickly clogged or gummed up.

Correspondence with Dr. Ray Brown and Stuart Mager at NCAT indicated they were experimenting with an ashing furnace and were planning on testing in mesh baskets. VTRC purchased a Thermolyne Model 30400 ashing furnace and began testing with a prototype version of the stainless steel sample baskets produced by QC Resource. The larger chamber dimensions (36 x 36 x 36 cm) and air injection manifold allowed the sample to be burned in the presence of oxygen while spread in a thin lift on a series of mesh baskets with a metal catch pan. The samples were heated at 135 °C in a forced draft oven until the sample was soft enough to be mixed, quartered and spread into a thin lift, typically 30-45 minutes. The sample was then spread in two thin lifts in a series of nested #20 stainless steel mesh baskets with a stainless steel drip pan placed under the baskets. The basket and sample weights were determined on an external scale. The assembly was placed in the ashing furnace set at 593°C (1100 °F). The airflow into the furnace chamber manifold was set at 20 l/min. Clean dry air was provided by an air compressor fitted with a refrigerated air dryer and pressure reducing valve. The sample burn time was determined by allowing a typical 1200 - 1500 g sample to burn for 35 minutes, removing it from the furnace and weighing it on an insulated external scale. The weight had to be determined as soon as the scale first stabilized because the weight increased as temperature decreased. The sample was then placed back in the furnace for an additional 5 minutes, at which time the weighing process was repeated. Due to the rapidly changing weight as the sample cooled it was difficult to determine an exact weight. When two consecutive readings displayed approximately the same weight, the first time was set as the burn time for future samples. Subsequent samples were prepared at the same sample mass used for time determination. In some instances, higher than optimum asphalt contents resulted in incomplete burns. These samples were returned to the furnace for an additional period of time, allowed to cool for 30 minutes, and weighed again. Though calibrations were developed for "hot" weights, the AC content determined from a cool sample was more accurate.

One set of nuclear specimens from the operator variance experiment was burned in the ashing furnace. Two SM specimens or one BM specimen were heated, mixed and quartered. One quarter was tested in the ashing furnace and the second was used for reflux extraction.

VTRC was provided with one of the 12 prototype NCAT ignition furnaces for round-robin testing as part of NCAT's study. The Barnstead/Thermolyne NCAT ignition furnace differs from the previous ashing furnace. First, it has an internal scale which measures the sample weight every 5 seconds during the test. A "temperature compensation" algorithm was developed by Barnstead/Thermolyne to account for the change in observed weight of the sample baskets as they change temperature from 25 to 538 °C. Data from the internal scale is sent to a controller which determines when a constant weight is achieved, at which time an audible alarm signals that the test should be terminated. The asphalt content of the sample is then calculated from the final weight using the temperature compensation factor. The controller also has provisions for the input of an aggregate correction factor. The aggregate correction factor accounts for the loss or breakdown of aggregate during ignition. One operator's SM specimens from the nuclear operator variance experiment were burned in the ignition furnace. Based on the results of the round-robin and the operator variance specimens a test methodology was developed and adopted for the 1995 construction season. *Virginia Test Method for Determination of Asphalt Content From Asphalt Paving Mixtures By the Ignition Method,* designation VTM 102, is presented in Appendix A.

Field Verification

In the fall of 1994, a Superpave™ Level I mix design was placed on Route 7 near Leesburg, Virginia. The test section was placed as part of National Cooperative Highway Research Program Project 9-7. During production, the ashing furnace, nuclear gauge with sample pans, nuclear gauge with sample plugs, and reflux extraction were used for asphalt content and gradation control. Samples were retained for testing in the NCAT ignition furnace. Cold-feed gradation samples were also taken.

RESULTS AND DISCUSSION

Nuclear Asphalt Content Gauge with Sample Plugs

Count Time

Samples were prepared at 4 asphalt contents with Shadwell Greenstone. The asphalt contents were ±0.7% and ±0.2% of optimum. Each pair of 100 mm specimens was tested at 1, 4, and 8 minute count time periods (This produced test times of 4, 16, and 32 minutes). Three replicate tests were performed for each count time period. The percent of asphalt was subtracted from the gauge reading for each of the 36 readings to determine the percent difference from the actual asphalt content (bias). The differences were sorted by asphalt content and test time, and an analysis of variance (ANOVA) was performed using Minitab Statistical Software and the general linear models (GLM). Table 4 summarizes the results of the ANOVA procedure. The results show that the differences between the mean bias for both the asphalt contents and count (test) times are not significant for the surface mix specimens.

The standard deviation is significantly larger for the 1 minute count time readings. However, the mean bias is smaller for the 1 minute count time readings. The sum of the variance (standard deviation squared) and the square of the bias is called the mean squared error (MSE). This provides a good indication of the better test procedure (count time). The results of the mean squared error calculations are presented in Table 5.

Table 4.
ANOVA Results: Differences between Gauge Readings and Actual Asphalt Contents

Factor	Result[1]	Prob. > F[2]
Surface Mix		
AC Content	NS	0.550
Count Time	NS	0.147
AC Content*Count Time	NS	0.122
Base Mix		
AC Content	S	0.010
Count Time	NS	0.787
AC Content*Count Time	NS	0.218

[1] NS- Not significantly different at the 5% level.
S - Significantly different at the 5% level.

[2] Probability of obtaining an F-value as large as the one computed if the factor level means are actually equal. A probability less than 0.05% indicates a significant difference.

Table 5.
Mean Squared Error Calculations Comparing 1, 4, and 8 Minute Count Time Periods

Count Time Minutes	Mean Bias[1] AC, %	Standard Deviation AC, %	MSE
Surface Mix			
1	-0.0142	0.1915	0.0369
4	0.0625	0.0883	0.0117
8	0.0875	0.1041	0.0185
Base Mix			
1	0.0067	.7782	0.0140
4	0.0207	.0737	0.0059
8	0.0027	.0591	0.0035

[1] Mean Bias = The average of the difference between the measured asphalt contents and the actual asphalt contents for set of count time replicates.

For the surface mix specimens, the 4 minute count time period had the lowest average MSE. For the base mix, the 8 minute count time period had only a marginally lower MSE than the 4 minute count time period. The 4 minute period was chosen to be used for the remainder of the testing for both mixes.

Specimen Weight Analysis

Samples were prepared at the design asphalt content. The weight of a pair of surface mix samples was varied ±10 g and ±25 g from the mean weight of the pairs of calibration specimens. The pairs of samples were also prepared at the same weight as the calibration specimens. Each pair of samples was tested 5 times (5 non-destructive replicates). A one-way ANOVA showed that at the 5% risk level there was no significant difference ($P = 0.726$) in the mean measured asphalt contents. However, the measured asphalt content appeared to increase with increasing sample weight. Therefore, a mean squared error analysis was performed. The results are presented in Table 6.

Results from Table 6 indicate that weight change does cause differences in standard deviation and MSE. Based on the low MSE and standard deviation of the 0 g difference specimens, it was decided to target a difference of ±5 g from the calibration specimen's weight. Smaller weight differences seemed impractical. This range was used for the remainder of the study.

Table 6.
Mean Squared Error Calculations for Surface Mix Specimens of Varying Weight

Weight Difference (g)	Mean Reading AC, %	Bias[1] AC,%	Standard Deviation AC, %	MSE
-25	4.49	-0.01	0.13	0.0164
-10	4.51	+0.01	0.16	0.0246
0	4.56	+0.06	0.07	0.0085
+10	4.53	+0.03	0.19	0.0380
+25	4.61	+0.11	0.16	0.0383

[1] The actual asphalt content was 4.5% for all of the specimens.

Since an unbalanced design was used for the base mix specimens, the analysis methods were altered. Base mix specimens were prepared at approximately -25, -10, 0, +15 and +25 g from the mean weight of the calibration specimens. Multiple specimens were prepared at -10 and 0 g difference. Each specimen was tested three times to produce a total of 30 test results. A regression analysis was performed using Minitab Statistical Software measured asphalt content and weight difference as predictor variables for actual asphalt content. Actual specimen weight differences were used for the analysis. A strong correlation was found between measured and actual asphalt content (the coefficient of determination (R^2) =0.971, the root mean square error, an estimate of the standard deviation of the regression line (RMSE) = 0.0784). The inclusion of the specimen's weight difference provided no improvement in the model (R^2 = 0.972, RMSE = 0.0786). The model was

$$AC,\% = 0.395 + 0.899 MeasuredAC\% - 0.00102 WTDiff.$$

where,

Measured AC, % = the asphalt content determined from a calibrated gauge.
WT Diff. = The weight difference between the gauge calibration specimens and the tested specimen.

Based on this model, a 25 gram difference would produce a 0.026% error in the asphalt content. The probability that the regression coefficient for weight difference (p value) was actually 0 was calculated at 0.371. It appears that typical variation in the specimen weight of 150 mm specimens would not cause a significant difference in measured asphalt content. In retrospect the experiment should have been conducted over a larger range of weight difference.

It should be noted that if the regression constant intercept was set to 0, the slope of the regression line between measured and actual asphalt content was 0.99 for the base mix specimens in the weight study with RMSE = 0.090. R^2 is difficult to interpret in this situation (constant = 0).

Effect of Aggregate Type and Operator Variance

To analyze the data, which included different design asphalt contents for each aggregate type, the actual asphalt content was subtracted from each gauge reading to produce a difference (bias). The difference was used for each of the 108 observations representing 3 operator, 3 aggregate types, 4 asphalt contents for each aggregate type, and 3 replicate readings for each pair of surface mix specimens tested. The same procedure was used for the 144 observations representing 3 operator, 4 aggregate types, 4 asphalt contents for each aggregate type, and 3 replicate readings for each pair of base mix specimens tested. Analysis of variance (ANOVA) (balanced model) was performed with the Minitab computer program to determine if operator, aggregate, or asphalt content had a significant effect on the gauge readings.

Table 7 summarizes the results of the ANOVA procedure, showing that the differences between operators, aggregates, and asphalt contents are significant, evidenced by the low probability of obtaining an F-value as large as the one computed. The interactions are also significant between operator and aggregate source and operator, aggregate source and AC content. Thus it may be expected that the variance of the gauge readings will be effected by operator, aggregate type, and asphalt content. The significance of aggregate source confirms the results of Burati et al. (1987). The significance of asphalt content within a given aggregate source suggests that the bias of the gauge changes as the measured asphalt content moves further from the center of the calibration. This is not surprising, since a three point calibration which produces a straight line regression equation was used. It is difficult to ascertain if the cause of operator variance is in the preparation of the test samples or in the operation of the gauge. In retrospect, one person should have mixed all of the test specimens with the operators compacting their own specimens, or each operator should have tested the same set of specimens.

To evaluate the testing variability, the standard deviation was calculated for each cell. The pooled standard deviation was calculated for each aggregate source by operator. Though three different operators were used, only one gauge was used so it is only possible to evaluate the within-lab standard deviation. The bias was calculated in a similar manner. The results for the surface mix specimens are reported in Table 8. The overall within-lab standard deviation for the surface mixes was 0.089% with an average bias of 0.069%. The absolute value of the average error was 2.51% (of the actual asphalt content) for the surface mix specimens with a standard deviation of 1.85%. The error was calculated by dividing the bias for each reading by the actual asphalt content. The absolute value of the error for each specimen was then averaged to produce the absolute value of the error for the group of specimens. The results for the base mix samples were calculated in the same manner and are reported in Table 9. The overall within lab standard deviation for the base mixes was 0.047% with an average bias of 0.080%. The absolute value of the average error was 2.98% (of the actual asphalt content) with a standard deviation of 2.07%. Thus, on average, a sample with 5.00% AC would be expected to have 0.15% AC error in the gauge asphalt content measurement.

Table 7.
Results of ANOVA on Differences between Gauge Readings and Actual Asphalt Contents Considering Operator, Aggregate Source, and Asphalt Content.

Factor	Surface Mixes		Base Mixes	
	Result[1]	Prob. > F[2]	Result[1]	Prob. > F[2]
Operator	S	0.000	S	0.000
Aggregate Source	S	0.001	S	0.000
AC Content	S	0.002	S	0.002
Operator * Aggregate Source [3]	S	0.000	S	0.000
Operator*AC Content [3]	NS	0.383	S	0.000
Aggregate Source * AC Content [3]	NS	0.150	S	0.000
Operator*Aggregate Source*AC Content [3]	S	0.000	S	0.000

[1] NS- Not significantly different at the 5% level.
S - Significantly different at the 5% level.

[2] Probability of obtaining an F-value as large as the one computed if the factor level means are actually equal.

[3] Interaction terms between the variables shown.

Table 8.
Pooled Standard Deviations and Bias by Aggregate Type and Operator for Surface Mix Specimens

Aggregate Source	Operator			Mean
	1	2	3	
Pooled Standard Deviation, AC, %				
1	0.062	0.108	0.075	0.082
2	0.054	0.107	0.126	0.096
3	0.144	0.052	0.074	0.090
Bias, AC, %				
1	0.062	0.011	0.151	0.075
2	0.237	0.032	0.064	0.111
3	0.001	-0.031	0.095	0.022

Table 9.
Pooled Standard Deviations and Bias by Aggregate Type and Operator for Base Mix Specimens

Aggregate Source	Operator			Mean
	1	2	3	
Pooled Standard Deviation, AC, %				
1	0.047	0.043	0.036	0.042
2	0.051	0.041	0.059	0.050
3	0.052	0.067	0.032	0.050
4	0.020	0.058	0.061	0.046
Bias, AC, %				
1	0.045	0.062	0.128	0.078
2	0.113	0.112	0.208	0.144
3	-0.029	-0.042	0.046	-0.008
4	0.148	0.094	0.072	0.106

Effect of Asphalt Source

At the initiation of the project, sufficient quantities of AC-20 and 30 were sampled to complete the testing. During the analysis stage, it was suggested that specimens should have been tested at the optimum asphalt content for each mix. Unfortunately, the binder supply had been depleted, though sufficient aggregate remained. New samples of AC-20 and 30 were obtained from the original supplier and samples were prepared at the optimum asphalt content. The original operators and calibrations were used to test the samples. In each mix, a shift in the bias of the readings was observed. A typical result is illustrated in Figure 1. A summary comparison of the average bias for the original and new surface mix specimens by aggregate type is presented in Table 10. The average "shift" in bias is 0.25%. This is alarming since many HMA contractors change their asphalt source on a weekly basis, based on price fluctuations. This would tend to indicate that a new calibration would be required for changes in binder source and changes within a given binder source with time.

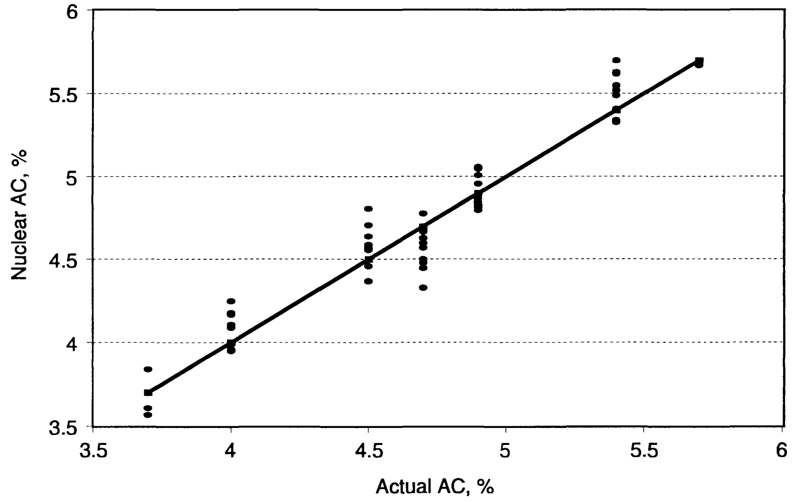

Figure 1. Typical shift in the bias of mix readings.

Table 10.
Comparison of Average Bias by Aggregate Type for Original and "New" AC-30

Surface Mix Aggregate Source	Average Bias (%AC) For All Operators	
	Original AC-30	"New" AC-30
1	+0.08	-0.14
2	+0.11	-0.13
3	+0.02	-0.28

Cross-Calibration of Nuclear Gauges with Sample Plugs

The original intent of the nuclear asphalt content gauge study was to develop a test method and conduct a round-robin to determine the within-lab and between-lab standard deviation and tolerances so the gauge could be used for asphalt content determination in VDOT's Volumetric Quality Index Specification. For this to be practical, the gauge calibration for each mix design had to be transferable between the contractor's and the Department's gauges. The cross-calibration procedure successfully used with the sample pans (Hughes 1994) had never been used with the sample plugs.

The cross-calibration procedure requires that a minimum of 5 samples be tested in each gauge with a spread in asphalt content at least 0.5% above and below the range of expected contents to be tested in the two gauges. For this study, cross-calibration for the surface mixes was performed with aggregate source #1, with pairs of Marshall samples produced at asphalt contents of 3.5, 4.2, 5.2, 6.2 and 7.2 %. Some alteration of the percent passing the 0.075 mm (#200) sieve was required to accommodate this range. A fit coefficient of 0.999 was obtained with the "master" gauge.

The calibrations for the surface mixes were then transferred to the cross-calibrated or "field" gauge. Operator #2's specimens for aggregate sources 1 and 2 were tested (3 non-destructive replicates) using the calibrations derived from the cross-calibration. Table 11 presents a comparison between the measurements in the master and field gauge. The bias, standard deviation, and absolute testing error are significantly higher for the field gauge measurements. Based on this limited experience, and the success of the concurrent ignition furnace testing, the plans for a round-robin with the nuclear gauge were abandoned. Possibly a better way to perform the initial cross-calibration could be developed to improve the results of the field gauge.

Table 11.
Pooled Standard Deviations, Bias, and Absolute Error Comparison between Master and Field Gauge for Specimens from Operator 2

Aggregate Source	Pooled Standard Deviation AC, %		Bias AC, %		Absolute Error (% of Asphalt Content)	
	Master	Field	Master	Field	Master	Field
1	0.108	0.198	0.011	0.424	3.68	9.25
2	0.107	0.342	0.032	0.353	2.29	8.32

Asphalt Content by the Ignition Method

After the completion of the nuclear asphalt testing, the retained operator variance surface mix samples were tested in three successive generations of ignition furnaces. Reflux extractions were run on splits of two operators' samples. All of the retained base mix specimens were tested in the NCAT (Thermolyne) ignition furnace.

Research conducted by NCAT and others indicated that a correction factor was needed with the ignition method to account for aggregate loss. A calibration procedure was not established for the muffle furnace since it was difficult to completely burn off the asphalt.

Calibration factors were developed for each aggregate type tested in the ashing furnace by burning four samples at the design asphalt content and determining the average difference between the measured and the actual asphalt content. The first sample tested was used to determine the burn time required to completely ignite the asphalt. A mixture sample weighing approximately 1200 g was placed in the ashing furnace at 593 °C (1100 °F) for 35 minutes, then removed from the furnace and weighed on an external scale. The scale's load cell was protected with a ceramic insulating plate. The weight was recorded when the scale first stabilized, before the weight changed as the sample cooled. The sample was then returned to the furnace for 5 minutes, removed, and weighed again. This process was continued until the difference between two consecutive readings was less than 1.0 g. The total elapsed time until the second-to-last reading was used for all future burns with that mix, unless asphalt residue was visible on the aggregates.

As the samples cooled, they gained weight. A weight change of approximately 10 g was typical as the sample and basket assembly cooled from 593 to 25 °C. It was initially believed that this might result from the sample taking on moisture from the room. However, the same phenomena was observed with empty stainless steel baskets. Since calibrations obtained with the room temperature weight produced smaller correction factors, calibrations were calculated by both methods for two of the aggregate sources.

The NCAT (Thermolyne) ignition furnace was calibrated by both an "aggregate only" procedure developed by NCAT for the round-robin (Brown, 1996) and by burning the original nuclear gauge calibration cores for each aggregate type. The aggregate-only samples were burned for 40 minutes at 575 °C (1067 °F) to simulate the typical time and temperature of a mixture test. The higher temperature (only 538 °C was used for mixture samples) simulated the ignition of the asphalt cement. The calibration cores provided 6 samples, 2 each at optimum and ±1% of optimum. Table 12 is a summary of the calibration factors for both furnaces. The mixture calibration produced from the nuclear gauge calibration cores was used in all cases for the data that follows.

Table 12.
Ignition Furnace Calibration Factors by Aggregate Type for Surface Mix Specimens

Aggregate Source	Ashing Furnace (593 °C)		Thermolyne Ignition Furnace (538 °C)	
	Weighed Hot	Weighed Cold	Mix	Aggregate Only
1	-1.13	-0.31	-0.09	-0.34
2	-1.49	NA	-0.45	-0.53
3	-1.00	-0.22	-0.06	-0.30

A summary of the ignition furnace testing, reflux extractions and nuclear asphalt content testing with sample plugs for the surface mix specimens is presented in Table 13. The standard deviation was calculated on the difference between the measured and actual asphalt contents. The results were not pooled, but calculated by test method. The results from the ashing furnace where the cooled sample were weighed on an external scale appear to be the best, but this is based on an extremely limited sample size (n=5). The second best method based on absolute % error and MSE is the Thermolyne ignition furnace. This furnace requires less technician labor and is not dependent on technician judgement for determining burn time. All of the methods with the exception of the muffle furnace are more accurate than reflux extraction. The ignition methods have the added advantage of producing a clean aggregate sample for gradation analysis.

Table 13.
Summary Statistics by Test Method for Surface Mix Specimens

Test Method	Number of Observations (n)	Standard Deviation, AC, %	Bias AC, %	Absolute % Error	MSE
Reflux Extraction	23	0.290	-0.113	5.16	0.0969
Nuclear Gauge	108	0.140	0.070	2.51	0.0245
Muffle Furnace	12	0.203	-0.439	9.09	0.2339
Ashing Furnace (HOT)	10	0.152	-0.007	2.51	0.0232
Ashing Furnace (COLD)	5	0.057	0.006	1.12	0.0033
Ignition Furnace	24	0.110	-0.033	1.50	0.0132

Mixture calibrations were performed on the base mix specimens tested in the Thermolyne ignition furnace. Table 14 shows the calibration factors. Interestingly, the limestone aggregate (source #4) has a positive correction factor, since the measured result is less than the actual asphalt content. This indicates that the aggregate gained weight during ignition. Analysis of Variance (General Linear Model) was performed on the base mix data using Minitab computer software. Table 15 summarizes the results of the ANOVA procedure. The results show that the differences resulting from aggregate type, operator, and asphalt content are not significant at the 5% confidence level.

Since ANOVA indicated no significant differences between aggregate source, operator, or asphalt content, a standard deviation of the differences between the measured and actual asphalt content was calculated using all 46 observations (2 missing samples). For comparison, the standard deviation of the nuclear gauge data was calculated the same way. The summary statistics for both methods are reported in Table 16.

Table 14.
Ignition Furnace Calibration Factors by Aggregate Type for Base Mix Specimens

Aggregate Source	Mix Calibration Factor (AC, %)
1	-0.15
2	-0.52
3	-0.06
4	+0.05

Table 15.
Results of ANOVA on Differences between Measured and Actual Asphalt Contents Considering Operator, Aggregate Source, and Asphalt Content

Factor	Result	Probability > F
Aggregate Source	NS	0.099
Operator	NS	0.920
Asphalt Content	NS	0.587

Table 16.
Summary Statistics by Test Method for Base Mix Specimens

Test Method	Number of Observations (n)	Standard Deviation AC, %	Bias AC, %	Absolute % Error	MSE
Nuclear Gauge	144	0.139	0.080	2.98	0.0257
Ignition Furnace	46	0.101	0.027	1.66	0.0109

Again, the statistics indicate that the ignition furnace is more accurate than the nuclear gauge, with the mean squared error of the nuclear gauge almost 2.5 times higher than the ignition furnace. Additionally, the variability of the ignition furnace is statistically not affected by aggregate source, operator, or asphalt content. The low bias agrees with the results of the round-robin conducted by NCAT (Brown, 1996).

Field Verification

The asphalt content of samples collected from the Route 7 project was determined in the field with a nuclear gauge using sample pans and the ashing furnace. Samples were also tested in the laboratory using the Thermolyne ignition furnace and reflux extraction. Washed gradations were determined from cold-feed samples, reflux extractions, ashing furnace samples, and ignition furnace samples.

All four methods of asphalt content determination tracked well, as shown in Figure 2. Note that the target asphalt content was lowered from 5.7% to 5.5% between the second and third samples. The nuclear gauge results were obtained by FHWA and are only available for the first 9 samples. No aggregate correction factor was needed for either the ashing or ignition furnaces. Since reflux extraction was the method used for acceptance tests at the time the samples were taken, T-tests and F-tests were performed comparing the samples' means and variances of the other methods to the reflux extraction results. FHWA's nuclear gauge results were excluded due to missing samples. Table 17 contains the sample means, standard deviations, T-test and F-test results. Neither the means of the alternate test methods or the variances were significantly different from reflux extraction at the 5% risk level.

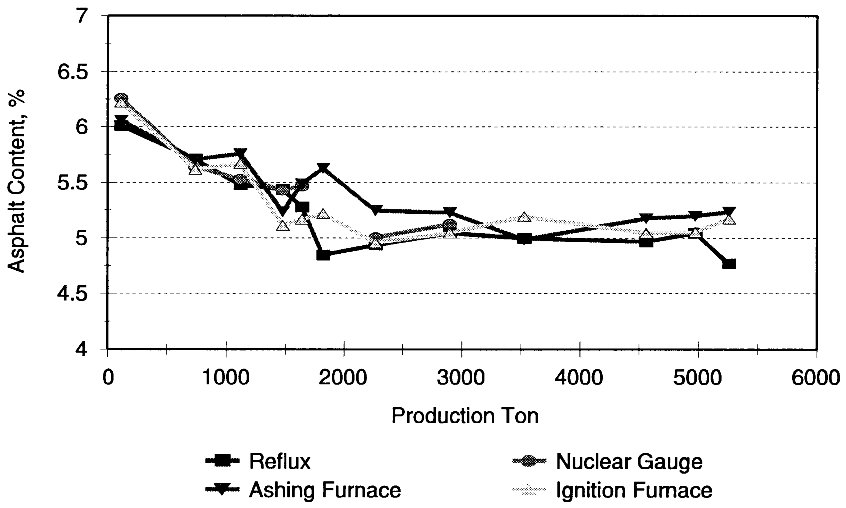

Figure 2. Comparison of four methods of asphalt content determination.

Table 17.
Results of T-Tests and F-Tests for Mean Differences between Alternate Test Methods and Reflux Extraction for Route 7 Field Data Asphalt Contents

Test Method	n	Mean (%)	Standard (%)	T-Test		F-Test	
				Result	Prob > |T|	Result	Prob > |F|
Reflux	12	5.21	0.378	NA	NA	NA	NA
Ashing Furnace	12	5.42	0.313	NS	0.17	NS	0.27
Ignition Furnace	12	5.29	0.367	NS	0.61	NS	0.46

Cold-feed gradations were taken to correspond to the nuclear gauge samples. Figure 3 shows the trends for the 9.5 mm sieve. Though the data appears to be more variable, Table 18 indicates that the results were not statistically different over the life of the project based on T-tests on the sample means.

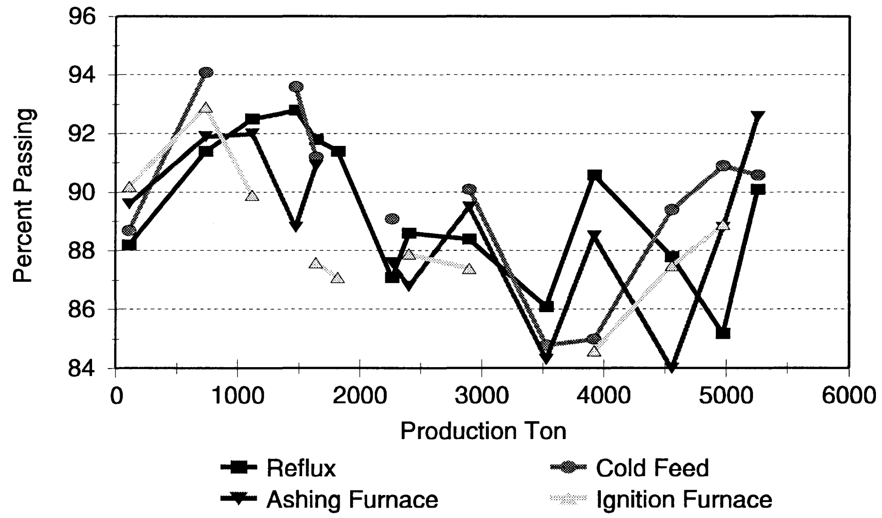

Figure 3. Trends for the 9.5 mm sieve.

Table 18.
Results of T-Tests Comparing Mean Differences between Alternate Test Methods and Reflux Extraction for Route 7 9.5 mm Sieve Data

Test Method	n	Mean (%)	Standard (%)	T-Test	
				Result	Prob > |T|
Reflux	14	90.1	2.81	NA	NA
Cold-Feed	11	89.8	2.94	NS	0.75
Ashing Furnace	13	88.9	2.72	NS	0.24
Ignition Furnace	11	88.75	2.41	NS	0.19

The trends for the 4.75 mm sieve are presented in Figure 4. Again the data appears variable, but the results in Table 19 indicate the percent passing was not statistically different over the life of the project based on T-tests on the sample means.

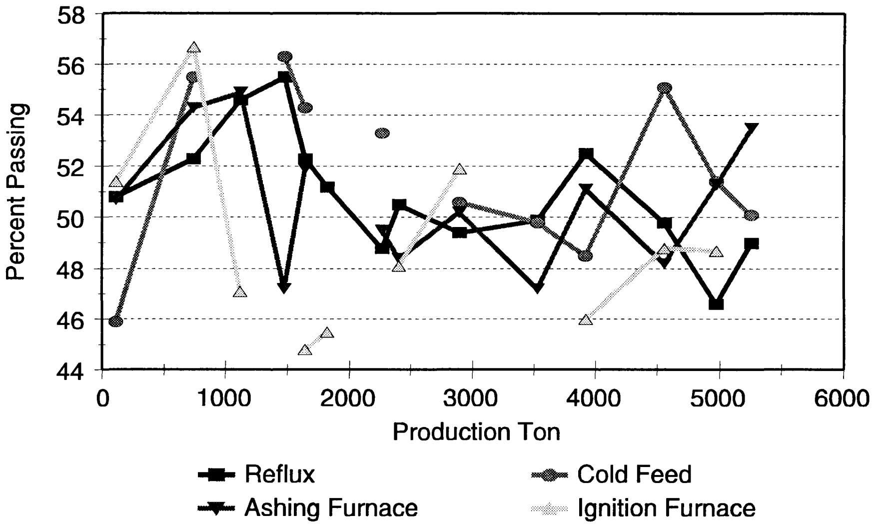

Figure 4. Trends for the 4.75 mm sieve.

Table 19.
Results of T-Tests Comparing Mean Differences between Alternate Test Methods and Reflux Extraction for Route 7 4.75 mm Sieve Data

Test Method	n	Mean (%)	Standard (%)	T-Test	
				Result	Prob > |T|
Reflux	14	50.9	2.36	NA	NA
Cold-Feed	11	51.9	3.28	NS	0.43
Ashing Furnace	13	50.7	2.55	NS	0.76
Ignition Furnace	11	49.4	3.85	NS	0.27

The trends for the 0.075 mm sieve are presented in Figure 5. The cold-feed gradations appear to often have a lower percent passing than the other methods. This may be due to breakdown in the drum creating additional P0.075 material. The results of the T-tests comparing the sample means presented in Table 20 indicate that the P0.075 contents determined from the cold-feed gradations are significantly different than the reflux extraction values at the 5% risk level. The ashing furnace results are also lower, but not significantly so. This may be the result of loss of fines due to the pressurized air flow from the manifold inside the chamber.

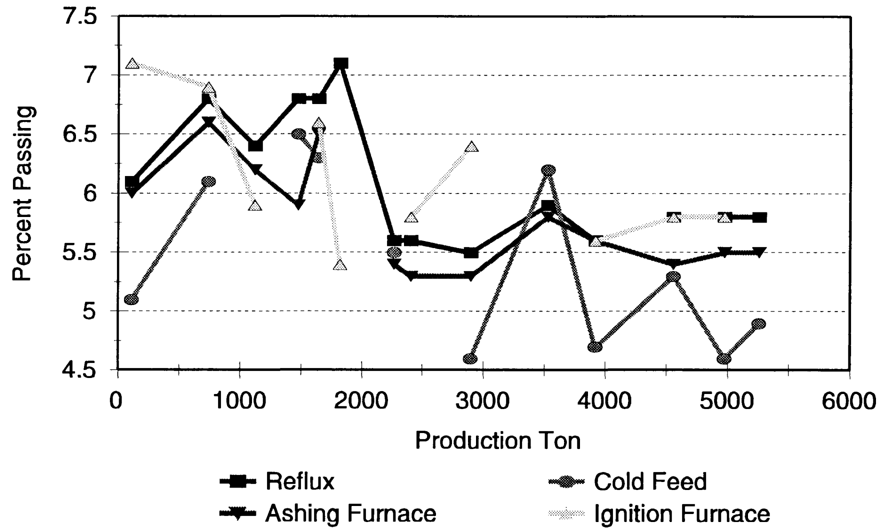

Figure 5. Trends for the 0.075 sieve.

Table 20.
Results of T-Tests Comparing Mean Differences between Alternate Test Methods and Reflux Extraction for Route 7 0.075 mm Sieve Data

Test Method	n	Mean (%)	Standard (%)	T-Test	
				Result	Prob > |T|
Reflux	14	6.11	0.553	NA	NA
Cold-Feed	11	5.44	0.726	S	0.019
Ashing Furnace	13	5.77	0.444	NS	0.086
Ignition Furnace	11	6.14	0.550	NS	0.92

Statistically, the ashing and ignition furnaces produced similar gradations to reflux extraction over the life of the project. However, the aggregate used for this project was a very hard traprock. Softer aggregates may suffer degradation in the ignition furnace.

CONCLUSIONS

1. The accuracy of the nuclear gauge with sample plugs is affected by the aggregate type, operator, and asphalt content of the specimen when a three point calibration is used.

2. When properly calibrated, the nuclear gauge with sample plugs, ashing furnace, and ignition furnace are all more accurate than reflux extraction.

3. The gradations performed on samples tested in the ignition furnace closely match those obtained by reflux extraction.

4. The ignition furnaces would be preferable to the nuclear gauge because:

   - It produces a clean aggregate sample for gradation analysis.
   - The accuracy of the furnace does not appear to be dependent on aggregate type or asphalt content.
   - Results from round-robin testing conducted by NCAT (Brown, 1996) indicate that calibrations are transferable from one furnace to another, whereas calibration transfer does not appear to work with the sample plugs in the nuclear gauge.

5. The ignition furnace with an internal weighing system is safer than the ashing furnace, since it requires less handling of the hot samples. The ignition furnace also eliminates errors which may be introduced due to the change in weight with changing temperature.

RECOMMENDATIONS

1. VTM 102: *Asphalt Content Determination of Hot Mix Asphalt Paving Mixtures By the Ignition Furnace* (Appendix A) should be implemented as the replacement method for reflux extraction.

2. Research should be continued with the ignition furnace to study the difference between aggregate-only and mixture calibrations as well as calibrations for mixtures containing RAP.

3. The physical properties of aggregate tested in the ignition furnace should be evaluated to see if they may be used in SUPERPAVE mix designs for the incorporation of RAP (to determine bulk specific gravity, absorption etc.).

ACKNOWLEDGMENTS

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Appendix A

Determination of Asphalt Content By The Ignition Method
VTM 102

Virginia Test Method
for
Determination of Asphalt Content from Asphalt Paving Mixtures by the Ignition Method

Designation: VTM 102

COMPLETE TEST METHODOLOGY

INDEX

Revised July 25, 1995

Virginia Test Method
for
Determination of Asphalt Content
from Asphalt Paving Mixtures
by the Ignition Method

Designation: VTM 102

1. **Scope**

1.1 This test method covers the determination of asphalt content of hot-mixed paving mixtures by ignition of the asphalt cement at 538°C (1000°F) in a furnace. The aggregate remaining can be used for sieve analysis using AASHTO Test Method T 30.

1.2 The values stated in metric units are to be regarded as the standard.

1.3 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. **Referenced Documents**

AASHTO Standards:

T 248	Reducing Field Samples of Aggregate to Testing Size
T 168	Sampling Bituminous Paving Materials
T 30	Mechanical Analysis of Extracted Aggregate

3. **Summary of Test Methods**

3.1 The asphalt in a sample of hot-mix paving material is burned by ignition at 538°C (1000°F). The asphalt content is calculated from the mass of ignited aggregate, moisture content, and temperature compensation for the change in mass of the sample container. The asphalt content is expressed as mass percentage of the moisture-free mixtures. This method may not be applicable to mixes containing fibers or ground tire rubber (dry process).

4. **Apparatus**

4.1 A forced air ignition furnace, capable of maintaining the temperature at 650°C (1200°F), with an internal balance thermally isolated from the furnace chamber accurate to 0.1 g. The balance shall be capable of weighing a 3,500 gram sample in addition to the sample baskets. The furnace shall calculate a temperature compensation factor for the change in weight of the sample baskets and provide for the input of a correction factor for aggregate loss. The furnace shall provide a printed ticket with the initial specimen weight, specimen weight loss, temperature compensation, correction factor, corrected asphalt content (%), test time, and test temperature. The sample chamber dimensions shall be at least 35.6x26.7x35.6 cm (WxHxD) (14x10.5x14 in). A method for reducing furnace emissions shall be provided. The furnace shall provide an audible alarm and indicator light when the sample weight loss does not exceed 0.02 percent of the total sample weight for two consecutive minutes. The furnace door shall be locked until the completion of the test procedure.

Note: The National Center for Asphalt Technology (NCAT) Asphalt Content Tester manufactured by Barnstead/Thermolyne and distributed by QC Resource (800) 296-7171 has been found to be suitable.

4.2 Two tempered stainless steel 2.36 mm (No. 8) mesh or otherwise perforated baskets, dimensions (LxWxH) 26.7x26.7x5.1 cm (10.5x10.5x2.0 in) with 5 cm (2 in) support legs. The baskets shall be nested. The baskets shall be provided with screening on the legs to confine the aggregate.

4.3 One stainless steel catch pan, dimensions (LxWxH) of 28.0x28.0x2.6 cm (11.0x11.0x1.0 in).

4.4 Oven capable of maintaining 125 ±5°C (257 ±9°F).

4.5 Balance, 8 kg or greater capacity, sensitive to 0.5 g for weighing sample in baskets.

4.6 Safety Equipment: safety glasses or face shield, high temperature gloves, and long sleeved jacket. Additionally, a heat resistant surface capable of withstanding 650°C (1202°F) and a protective cage capable of surrounding the sample baskets shall be provided.

4.7 Miscellaneous Equipment: pan dimensions (LxWxD) 38x38x5 cm (15x15x2 in) minimum for transferring samples after ignition, spatulas, bowls, and wire brushes.

5. **Sampling**

5.1 The test sample shall be the end result of quartering a larger sample taken in accordance with VTM-48 (AASHTO T 248 may be used as a guide to quartering). Note: VTM-48 is a modified version of AASHTO T 168.

5.2 Preparation of Test Specimens:

5.2.1 If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large flat pan and warm to 125°C ±5°C (257°F ±9°F) for 25 minutes. The sample shall not be heated for more than 1 hour.

5.2.2 The size of the test sample shall be governed by the nominal maximum aggregate size of the mixture and shall conform to the mass requirement shown in Table 1 (Note 1):

Note 1-When the mass of the test specimen exceeds the capacity of the equipment used, the test specimen may be divided into suitable increments, tested, and the results appropriately combined for calculation of the asphalt content (weighted average).

Table 1. Size of Sample

Nominal Maximum Aggregate Size (mm)	Sieve Size	Minimum Mass of Sample (g)
4.75	(No. 4)	1200
9.5	3/8 in.	1200
12.5	½ in.	1500
19.0	3/4 in.	2000
25.0	1 in.	3000
37.5	1 ½ in.	4000

Sample sizes should not be more than 400 g greater than the minimum recommended sample mass. Large samples of fine mixes tend to result in incomplete ignition of the asphalt.

5.2.3 In addition, a test specimen for moisture determination (VTM-49) will be made as deemed necessary. The specimen used for moisture determination may not be used for asphalt content determination.

6. Calibration

Two calibration procedures are provided. Either method may be required by the engineer. For mix designs containing RAP, sufficient quantity of RAP should be sampled such that the binder content of the RAP may be estimated, and to provide for the RAP to be used in the mix calibration. The binder content of the RAP will be estimated from the average of four samples (RAP only) burned in the furnace. The portions of RAP should be obtained using a sample splitter.

Certain aggregate types may result in an unusually high calibration factor and erroneous gradation results. Such mixes should be calibrated and tested at a lower temperature, typically 482°C (900°F) or as approved by the Engineer.

6A. Mixture Calibration, Method A

6A.1 This method may be affected by the type of aggregate in the mixture. Accordingly, to optimize accuracy, a calibration factor will be established with the testing of a set of calibration samples for each mix type. This procedure must be performed before any acceptance testing is completed.

6A.2 Two calibration specimens conforming to the mass requirements of Section 5.2.2 shall be prepared at the design asphalt content and at ± 0.5% of the design asphalt content for a total of six specimens. A butter mix shall be prepared at the design asphalt content, mixed and discarded prior to mixing any of the calibration specimens to ensure an accurate asphalt content. Aggregate used for the calibration specimens shall be sampled from stockpiled material produced in the current construction season. Any method may be used to combine the aggregates, but an additional "blank" specimen shall be batched and tested according to AASHTO T 30. The washed gradation shall fall within the JMF (mix design) tolerances.

6A.3 The freshly mixed specimens may be placed directly in the sample baskets. If allowed to cool, the samples must be preheated in a 125°C (257°F) oven for 25 minutes. Do not preheat the sample baskets.

6A.4 Preheat the ignition furnace to 538°C (1000°F) Record the furnace temperature (set point) prior to the initiation of the test.

6A.4 Enter a calibration factor of **<u>0.00</u>** in the ignition furnace.

6A.5 Weigh and record the weight of the two sample baskets and catch pan (with guards in place).

6A.6 Place the bottom sample basket in the catch pan. Evenly distribute approximately one half of the calibration specimen in the lower basket taking care to keep the material away from the edges of the basket.

6A.7 Place the upper sample basket on the bottom basket assembly. Evenly distribute the remaining specimen in the top basket. Use a spatula or trowel to level the specimen.

6A.8 Weigh and record the sample, baskets, catch pan, and basket guards. Calculate and record the initial weight of the sample specimen (total weight - the weight of the sample basket assembly).

6A.9 Input the initial weight of the sample specimen in whole grams into the ignition furnace controller. Verify that the correct weight has been entered.

6A.10 Open the chamber door and place the sample baskets in the furnace. Close the chamber door and verify that the sample weight (including the baskets) displayed on the furnaces scale equals the total weight recorded in Section 6.8 within ± 5g. Differences greater than 5 grams or failure of the furnace scale to stabilize may indicate that the sample baskets are contacting the furnace wall. Initiate the test by pressing the start/stop button. This will lock the sample chamber and start the combustion blower.

6A.11 Allow the test to continue until the stable light and audible stable indicator indicates the test is complete. Press the start/stop button. This will unlock the sample chamber and cause the printer to print out the test results.

6A.12 Open the chamber door, remove the sample baskets and allow to cool to room temperature (approx. 30 minutes).

6A.13 Perform a gradation analysis on the residual aggregate as indicted in Section 8.

6A.14 Once all of the calibration specimens have been burned, determine the difference between the actual and measured asphalt contents for each sample. The mix calibration factor is the average of the measured differences.

Alternate Calibration Procedure:

6B. <u>Aggregate Only Calibration, Method B</u>

6B.1 This method may be affected by the type of aggregate in the mixture. To optimize accuracy, a calibration factor will be established with the testing of a set of calibration samples for each mix type. This procedure must be performed before any acceptance testing is completed.

6B.2 Four blank calibration specimens (aggregate and RAP if applicable) conforming to the mass requirements of Section 5.2.2 shall be prepared to meet the job-mix formula (JMF). Aggregate used for the calibration specimens shall be sampled from stockpiled material produced in the current construction season. Any method may be used to combine the aggregates, however an additional "blank" specimen shall be batched and tested according to AASHTO T 30. The washed gradation shall fall within the JMF tolerances.

6B.3 Preheat a specimen in a 125°C (257°F) oven for 20 minutes. thoroughly mix the specimen with a spatula prior to introducing it into the sample baskets.

6B.4 Preheat the ignition furnace to **<u>575°C (1067°F.)</u>** Record the furnace temperature set point prior to the initiation of the test.

6B.4 Enter a calibration factor of **<u>0.00</u>** in the ignition furnace.

6B.5 Weigh and record the weight of the two sample baskets and catch pan (with guards in place).

6B.6 Place the bottom sample basket in the catch pan. Evenly distribute approximately one half of the calibration specimen in the lower basket taking care to keep the material away from the edges of the basket.

6B.7 Place the upper sample basket on the bottom basket assembly. Evenly distribute the remaining specimen in the top basket. Use a spatula or trowel to level the specimen.

6B.8 Weigh and record the specimen, baskets, catch pan, and basket guards. Calculate and record the initial weight of the sample specimen (total weight - the weight of the sample basket assembly).

6B.9 Input the initial weight of the sample specimen in whole grams into the ignition furnace controller. Verify that the correct weight has been entered.

6B.10 Open the chamber door and place the sample baskets in the furnace. Close the chamber door and verify that the sample weight (including the baskets) displayed on the furnaces scale equals the total weight recorded in Section 6.8 ± 5 g. Differences greater than 5 grams or failure of the furnace scale to stabilize may indicate that the sample baskets are contacting the furnace wall. Initiate the test by pressing the start/stop button. This will lock the sample chamber and start the combustion blower.

6B.11 Specimen to remain in the furnace for: 1200-1500 g = 40 minutes, 2000-2400 g = 60 minutes, 3000 g or more = 75 minutes. The stable light and audible stable indicator may come on after 15-30 minutes; do not stop the test at this time. Press the start/stop button when the furnace timer indicates the appropriate times for the above different sample weights. This will unlock the sample chamber and cause the printer to print out the test results.

6B.12 Open the chamber door, remove the sample baskets and allow to cool to near room temperature (approximately 30 minutes).

6B.13 Perform a gradation analysis on the residual aggregate as indicted in Section 8.

6B.14 The mix calibration factor is calculated as follows:

$$ACA = \frac{\sum AC\%_{test1} + AC\%_{test2} + AC\%_{test3} + AC\%_{test4}}{4}$$

$$Mix\ Calibration\ Factor = ACA - \left(RAC\% \times \frac{\%RAP}{100}\right)^{1}$$

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[1] If RAP is used in mix.

where:

ACA = Aggregate Calibration Average
AC% = Corrected Asphalt Content for Aggregate Test
RAC% = Average RAP Corrected AC%

6B.15 The calibration should be repeated each time there is a change in the mix design or as requested by the engineer.

7. **Test Procedure**

7.1 Preheat the ignition furnace to 538°C (1000°F). Record the furnace temperature (set point) before beginning the test.

7.2 Enter the calibration factor for the specific mix to be tested as determined in Section 6 in the ignition furnace.

7.3 Weigh and record the weight of the two sample baskets and catch pan (with guards in place).

7.4 Prepare the sample as described in Section 5.2. Place the bottom sample basket in the catch pan. Evenly distribute approximately one half of the specimen in the lower basket taking care to keep the material away from the edges of the basket.

7.5 Place the upper sample basket on the bottom basket assembly. Evenly distribute the remaining specimen in the top basket. Use a spatula or trowel to level the specimen.

7.6 Weigh and record the sample, baskets, catch pan, and basket guards. Calculate and record the initial weight of the sample specimen (total weight - the weight of the sample basket assembly).

7.7 Input the initial weight of the sample specimen in whole grams into the ignition furnace controller. Verify that the correct weight has been entered.

7.8 Open the chamber door and place the sample baskets in the furnace. Close the chamber door and verify that the sample weight (including the baskets) displayed on the furnace scale equals the total weight recorded in Section 7.8 ± 5 g. Differences greater than 5 grams or failure of the furnace scale to stabilize may indicate that the sample baskets are contacting the furnace wall. Initiate the test by pressing the start/stop button. This will lock the sample chamber and start the combustion blower.

7.9 Allow the test to continue until the stable light and audible stable indicator indicate the test is complete. Press the start/stop button. This will unlock the sample chamber and cause the printer to print out the test results.

7.10 Open the chamber door, remove the sample baskets and allow to cool to room temperature (approximately 30 minutes).

8. Gradation

8.1 Allow the specimen to cool to room temperature in the sample baskets.

8.2 Empty the contents of the baskets into a flat pan. Use a small wire sieve brush to ensure that any residual fines are removed from the baskets.

8.3 Perform the gradation analysis according to AASHTO T 30.

9. Report

9.1 Always report corrected asphalt content, mix calibration factor, temperature compensation factor, total percent loss, sample mass, and test temperature. Attach the original printed ticket to the report. An example data sheet is attached.

10. Precision and Bias

10.1 Precision and Bias were determined in an NCAT Round-Robin study for surface mixes.

Asphalt Content	Standard Deviation, %	Acceptable Range of Two Test Results, %
Single-Operator Precision	0.04	0.11
Multi-laboratory Precision	0.06	0.17

Note: These precision statements are based on 4 aggregate types, 4 replicates, and 12 laboratories participating with 0 laboratory results deleted as outlying observations. All 4 aggregates were tested in surface mixes and had relatively low absorption values.