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Quantifying Antistrip Additives in Asphalt (Binder & Mixes)

by

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16. Abstract

In this study, litmus and colorimetric tests were developed to quantify the contents of amine-based antistrip additives in asphalt binders and mixes. In addition, the effect of prolonged heating on antistrip additive content was evaluated for both asphalt binders and mixes. Results of this study indicate that both litmus and colorimetric tests are capable of detecting and quantifying amine-based antistrip additives in asphalt binders and mixes. Also, both test methods were able to validate the antistrip additive contents in field samples with known additive contents.

When subjected to prolonged heating periods, the antistrip additive content decreased substantially for both asphalt binders and mixes. For asphalt binders, no antistrip additive content was detectable after 24 to 48 hours of extended heating; for mixes, the measured antistrip additive content approached zero percent after 6 to 12 hours of extended heating.

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Executive Summary

Stripping is a phenomenon of loss of bond or adhesion between the asphalt binder and the aggregate in asphalt mixes. Stripping frequently results from the presence of water, and most agencies require the use of antistrip additive to control moisture damage. NCDOT requires antistrip additive in all asphalt mixes to improve the performance of the pavement. However, there is no quick and convenient method or standard for detecting the presence and the amount or percentage of organic antistrip additive in asphalt binders or mixes. A reliable standardized test procedure will allow NCDOT to determine whether the specified level of organic antistrip additive has been added to the mix and hence, reduce the amount of substandard asphalt mix being placed in the field. It will also allow NCDOT and asphalt manufacturers to have a quality control test to inspect asphalt binders and hot mix samples.

The objective of this research was to develop a reliable and repeatable laboratory and/or field test procedure to determine the amount of organic antistrip additive in asphalt binders and asphalt mixtures. In this investigation, two test methodologies were investigated. These include: 1) a litmus test using the StripScan device and 2) a colorimetric test using amine extraction technique. Both methodologies utilize a solid-state spectrophotometer for the analysis of amine-based antistrip additive concentration in asphalt binders and mixes qualitatively and quantitatively.

Two types of amine-based antistrip additives were used in this study. Antistrip additive LOF 6500 is a modified fatty amidoamine, and Morlife 2200 is a mixture of alkyloxylated aliphatic polyamines, alkyl amines, and polyamines. The asphalt binders and mixes used in this study were obtained from NCDOT. Two aggregate types from different sources and with different gradations were used. Although, a PG 64-22 asphalt binder was used for all mixes, the asphalt binder used for each aggregate type was from a different source.

Results of this study indicate that both litmus and colorimetric tests are capable of detecting and quantifying amine-based antistrip additives in asphalt binders and mixes. Also, both test methods were able to validate the antistrip additive contents in field samples with known additive contents.

When subjected to prolonged heating periods, the antistrip additive content decreased substantially for both asphalt binders and mixes. For asphalt binders, no antistrip additive content was detectable after 24 to 48 hours of extended heating; for mixes, the measured antistrip additive content approached zero percent after 6 to 12 hours of extended heating.

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1. Introduction

1.1 Background and Literature Review

The surface course of highways must be tough enough to resist distortion under traffic and provide a smooth and skid-resistant riding surface; meanwhile, it must be waterproof to protect the entire pavement and subgrade from the weakening effect of water [1]. Pavement performance is determined by the strength of the adhesive bond between the asphalt binder and the aggregate [2]. Loss of adhesion at the asphalt-aggregate interface due to the invasion of water, commonly called stripping, is one of the principal events that lead to the failure of asphalt pavements [3]. Once initiated, stripping usually progresses rapidly. Widespread stripping can lead to strength loss, which causes cracking and raveling with the eventual formation of potholes. This result in shorter pavement life and many millions of dollars in pavement damage each year. A survey of state highway and other agencies found that stripping problems were widespread and influenced by a large number of factors including aggregate type, asphalt binder grade and source, mix design, construction, and climate [4]. Antistrip additives are commonly required for improving adhesion between the asphalt binder and the aggregate surface and, thus, the resistance of asphalt pavement to stripping damage. Antistrip additives include lime, fatty amines, and styrene-butadiene [5].

Amine-based antistrip additives are commonly required by state highway agencies for improving adhesion between the asphalt binder and the aggregate surface to increase the resistance of asphalt pavement to stripping damage. Just as the content of asphalt binder is a control index for asphalt mix design, the content of amine-based antistrip additive is important. However, the detection and quantification of the content of amine-based antistrip additive in hot-mix-asphalt (HMA) remains a practical problem. In addition there are concerns in the asphalt industry regarding the volatilization of these additives once they have been incorporated into the binders and mixes. Because a quick and convenient way of checking the amount of antistrip additives is lacking, the asphalt pavement material is not checked for the level of antistrip additive as often as it should be. As a result, the potential absence or variability in the dosage of antistrip additive, or volatilization of additive due to prolonged heating creates uncertainty in the quality of asphalt pavements.

One of the most commonly used procedures is the Tensile Strength Ratio (TSR) test, which is an indirect method to determine the presence of additives through measuring the performance of the asphalt mix. However, the TSR test method generally takes days for test results to be available, and in many instances it is unreliable. Tarrer et al. [6] has proposed a procedure that is a modified version of ASTM Test Method D 2073 [7] that allow direct measurement of the amount of amine-based antistrip additives in asphalt binder using a titration test. Ulrich et al. [8] further developed the titration test method so that it can be used for both asphalt binders and mixes. In this study, litmus and colorimetric test methods are presented. These methods are quick, simple, and reliable for quantifying amine-based antistrip additive contents in asphalt binders and mixes.

NCDOT requires antistrip additives in all asphalt mixes in order to increase the resistance of the mixes to moisture-induced damage. Absence or non-uniform use of the antistrip additive can lead to severe reduction in pavement life. Two such detrimental effects were noted recently in North Carolina? 1) failure to use antistrip additive in Rutherford County in 1998 [9] led NCDOT to require the contractor to put 1-inch additional surface mix over an asphalt pavement in which 25,000 tons of material had already been placed and compacted; 2) results of two NCDOT studies [10, 11] show that the severe delamination problems in Buncombe County are directly attributable to the moisture sensitivity of asphalt mixes containing baghouse fines.

In order to develop a reliable test procedure, two methodologies were evaluated in this study. These include: 1) a litmus test using the StripScan device and 2) a colorimetric test following the extraction of organic antistrip additive from asphalt binders and mixes.

1.2 Objectives and Scope of the Study

The principle objective of this study was to present a standardized, reproducible and quantifiable test procedure to detect organic antistrip additives in asphalt binders and asphalt mixes. A reliable and standardized test procedure would allow NCDOT to determine if the specified level of organic antistrip additive has been added to the asphalt binder or mix and hence, reduce the amount of substandard asphalt mix being placed in the field. It also allows NCDOT and contractors to have a quality control test to inspect asphalt binders and hot mix samples. In particular, the principal work tasks were as follows:

1. Provide a literature review to determine the current state of knowledge regarding detection of organic antistrip agents in asphalt binders and mixes.
2. Determine the ability and degree of complexity of the litmus test using StripScan to detect the presence and concentration of antistrip additives in asphalt binders and mixes.
3. Develop a technique for extracting antistrip additive from asphalt binders and mixes.
4. Determine the ability and degree of complexity of the colorimetric technique to detect the presence and concentration of antistrip additives in asphalt binders and mixes.
5. For both the colorimetric and litmus tests, investigate the accuracy and repeatability of the test.
6. Determine if litmus and colorimetric tests can quantify antistrip additives in field samples.
7. Determine the effect of prolonged heating on antistrip additive contents in asphalt binders and mixes using litmus and colorimetric tests.
8. Correlate test results from colorimetric and litmus tests.
9. Based on the results, recommend a standardized test procedure that can be adopted and followed by NCDOT.

The scope of this report is limited to the quantification of amine-based antistrip additives. Detection of inorganic additives, particularly lime, was not considered in this study. Figure 1-1 provides the work plan for the project.

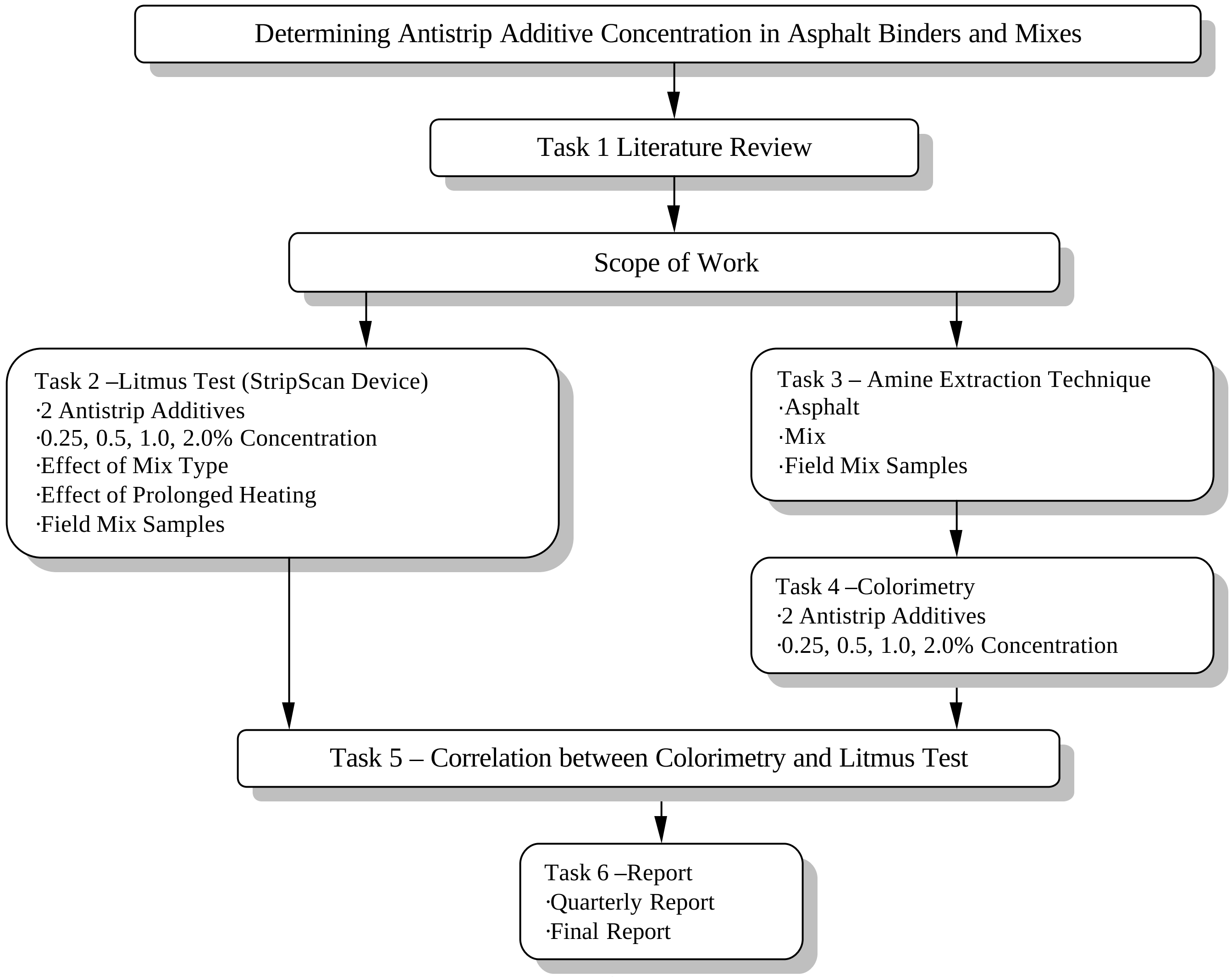


Figure 1-1 Summary of Research Approach and Methodology

2. Quantifying Antistrip Additive Using Litmus Test Method

2.1 Litmus Test Overview

A simple three-step procedure was developed to detect antistrip additives in asphalt binders and mixes. In the first step, the organic antistrip additive is volatilized by heating asphalt binders or mixes. In the second step, a litmus paper is exposed to the vapors containing the volatilized antistrip additive for a prescribed time period. In the third step, a spectrophotometer is used to measure the color change of the litmus paper that is brought about by the exposure to the antistrip additive. The difference in spectrophotometer readings of the litmus paper before and after vapor exposure is the color index that is used to quantify the amount of antistrip additive.

The litmus test is an intuitively simple procedure that at a minimum requires a device that can control the sample temperature at a desired level, and a spectrophotometer that can read the change in color (color index) of the litmus paper that results from exposure to the chemical vapors. In this study the StripScan device (InstroTek, Inc, Raleigh, NC, shown in Figure 2-1) was used to measure the quantity of antistrip additives in asphalt binders and mixes.

2.2 Materials

Most of the organic antistrip additives are proprietary chemical mixtures, and detailed characterizations are not usually available. Typically, organic antistrip additives contain fatty amines such as long-chain, primary, secondary and tertiary amines, diamines, amidoamines and imidoazolines [12]. Two types of additives were used in this research. LOF 6500 is a modified fatty amidoamine and Morlife 2200 is polyamine and mixed polycycloaliphatic polyamine, their physical properties are shown in Table 2-1.

Both of these antistrip additives were tested with PG 64-22 asphalt binder from the Citgo Wilmington terminal. Two types of field mixes were also tested. For both the field mixes, the job-mix-formula (JMF) required only LOF 6500 antistrip additive. However, during lab testing of the materials obtained from the field, both LOF 6500 and Morlife 2200 were used as antistrip additives. Details of the field mixes are as follows:

Material Type I:
Blend Percentages and Gradation are shown in Table 2-2 and Table 2-3.
Total Binder Content: 4.50%
Binder Grade: PG 64-22 Citgo Wilmington asphalt
Mix Temperature: 300°F
Antistrip Additive: LOF 6500 antistrip additive in the amount of 0.5%.

Material Type II:
Blend Percentages and Gradation are shown in Table 2-4 and Table 2-5.
Total Binder Content: 5.10%
Binder Grade: PG 64-22 Trumbull asphalt
Mix Temperature: 300°F
Antistrip Additive: LOF 6500 antistrip additive in the amount of 0.5%.

2.3 Procedure to Quantify Antistrip Additives in Asphalt Binders and Mixes

To determine the presence and amount of a given antistrip additive in the binder or mix, the spectrophotometer readings must initially be calibrated with asphalt binders or mixes of interest that contain known amounts of antistrip additive. Based on considerable experience developed during this study, it is suggested that 100-g asphalt binder samples or 2000-g asphalt mix samples be used for calibration and analysis of unknown samples.

2.3.1 Calibration procedure to determine antistrip additive content in asphalt binder

Initially, several tests were conducted to gain experience in using the StripScan device. The final test method used for producing the asphalt cement with antistrip additive was to take 500-g of the asphalt sample, adding to it a given additive percentage and mixing thoroughly so that the additive was uniformly dispersed. Immediately after mixing, the asphalt sample was divided into five 100-g batches to prevent segregation of the antistrip additive from the asphalt cement.

Based on the experience obtained in using the StripScan device, the following procedure was developed and followed for testing.

1. Based on the desired quantity of asphalt binder, weigh out the required mass of antistrip additive and place into a large metal can. For example, to obtain a 0.25% additive content in 500-g of asphalt binder, 1.25-g additive is weighed out. Subsequently, pour 500-g of heated asphalt into the can containing the measured additive content.
2. Mix thoroughly for at least 3 min. to ensure that the additive is uniformly distributed in the asphalt binder. A low-shear mixer of the type used for producing modified asphalt binders may be used.
3. Immediately after mixing, pour 100-g samples into individual test cans and close the can lid. In this study, 150-mm diameter cans were used as shown in Figure 2-2. It should be noted here that the can lid required for the litmus device has a 25-mm diameter opening in the middle to facilitate litmus testing. This hole should be sealed with a cork or rubber stopper during sample preparation to prevent vapor loss. At this point, the individual cans containing 100-g of asphalt binder with additive may be stored temporarily at room temperature.
4. Using steps 1 to 3, prepare samples with different levels of antistrip additive contents. In this study, five levels of antistrip additive content were evaluated? 0, 0.25, 0.5, 1.0 and 2.0% by weight of asphalt binder.

5. Immediately prior to testing, heat the sample in a forced draft oven at 150°C for 15 min. It should be noted that for multiple sample testing, the samples should not all be heated simultaneously. The sample heating process should be staggered based on operator efficiency such that while a given sample is being analyzed with the litmus test, the other samples are not being over-conditioned by heating them for more than 15 min.
6. Transfer the heated sample to the litmus device. A heating plate in the device maintains the sample temperature at 150°C as verified with a thermocouple introduced through a small hole in the can lid. At this point, the 25-mm diameter opening in the can lid should be unplugged.
7. A litmus test strip is brought into contact with the vapors escaping through the lid opening for a period of 3 min. It should be noted that before exposing the litmus strip to vapors, it has to be scanned with the spectrophotometer to establish a baseline reading.
8. After 3 min. of exposure to the vapors, the litmus strip is removed and immediately scanned with the spectrophotometer. The difference in spectrophotometer readings before and after vapor exposure is the color index that corresponds to the amount of antistrip additive present in the binder.
9. Repeat the procedure for replicate samples and for samples containing different levels of antistrip additive contents.
10. Establish a calibration curve (regression equation) between the additive content and the color index determined by the spectrophotometer.

In this study, steps 7 to 10 were fully automated in the litmus test using the StripScan device shown in Figure 2-1.

2.3.2 Calibration procedure to determine antistrip additive content in asphalt mixes

1. Incorporate the required amount of antistrip additive into asphalt binder. This process is the same as that presented above.

2. Prepare asphalt mix samples containing different antistrip additive contents. In this study, three 2000-g mix samples were prepared for each level of antistrip additive content.
3. Before testing, preheat the sample for 1-h. Leave the lid open when heating and stir (agitate) the sample every 15-30 min.
4. Follow steps 6 to 10 outlined above for the asphalt binder.

2.4 Test Results and Discussion

2.4.1 Test results for antistrip additive in asphalt binder

For the litmus test, PG 64-22 Citgo Wilmington asphalt binder samples were prepared using LOF 6500 and Morlife 2200 antistrip additives at levels of 0, 0.25, 0.5, 1.0 and 2.0% by asphalt binder weight. For each level of antistrip additive content, five replicate samples, each weighing 100-g, were prepared. Each sample was tested three times using different litmus strips. Tables 2-6 and 2-7 show the color index obtained using the StripScan device for PG 64-22 Citgo Wilmington asphalt binder containing the two antistrip additives LOF 6500 and Morlife 2200, respectively.

It should be noted that the calibration curves presented in Figures 2-3 and 2-4 for the two antistrip additives differ because of the different chemical compositions of the additives. Once established, these calibration curves can be used for quality control and quality assurance of field samples.

Figure 2-3 shows the correlation between the LOF 6500 additive content and the color index; the coefficient of determination (R^2) was 0.92. It can be seen from these data that the StripScan is fairly effective in detecting not only the presence of the antistrip additive LOF 6500 but also the amount (percentage) of the additive in the binder. That is, the StripScan is able to detect qualitatively as well as quantitatively the presence of the antistrip additive. Similarly, Figure 2-4 shows Morlife 2200 antistrip additive content as a function of color index. It should be noted that in Figures 2-3 and 2-4, for a given additive content, there is a fair amount of scatter in the measurement of color index. This scatter is accounted for in the regression R^2 values and will inherently affect the accuracy of predicted additive content values.

One difficulty that was encountered with the measurement of the Morlife 2200 antistrip additive was that the additive content could only be measured quantitatively up to a level of 0.5%. Beyond this limit the color index did not change measurably as is evident in Table 2-7. This behavior is attributed to saturation of the litmus paper. However, up to the 0.5%-level of antistrip additive content, the presented relationship has an R^2 value of 0.99. It needs to be noted that the exposure time of the litmus paper to the vapors was 3 min. in this study. For some antistrip additives such as Morlife 2200 that tend to saturate the litmus paper more quickly, a testing agency may need to reduce the exposure time, testing temperature, or both if high levels of antistrip additive need to be quantified in asphalt binders.

Once developed, the regression equations (calibration curves) shown in Figures 2-3 and 2-4 can be used to detect the presence and amount of antistrip additive in field asphalt binder samples for quality control and quality assurance purposes with a reasonable degree of certainty as indicated by the R^2 values (> 0.9). Although the quantitative determination of higher percentages of antistrip additive may be difficult for some additive types, the data show that the procedure developed herein can detect the presence of antistrip additive in a qualitative manner in such instances.

2.4.2 Test results for antistrip additives in asphalt mixes

Although the JMF requires the use of LOF 6500 antistrip additive for mixes I and II, testing was also conducted on mixes with Morlife 2200 antistrip additive.

Two types of asphalt mix samples were prepared using LOF 6500 and Morlife 2200 antistrip additives at levels of 0, 0.25, 0.5, 1.0 and 2.0% by asphalt binder weight. It should be noted that these two types of asphalt mix differ not only in gradation but also in asphalt type. The PG 64-22 Citgo Wilmington asphalt was used for asphalt mix I, whereas PG 64-22 Trumbull asphalt was used for asphalt mix II. For each level of antistrip additive content, three replicate samples, each weighing 2000-g, were prepared. Each sample was tested twice using different litmus strips. Tables 2-8 and 2-9 show the color index obtained from the StripScan device for asphalt mix I containing the two antistrip additives LOF 6500 and Morlife 2200, respectively. Similarly, Tables 2-10 and 2-11 show color index data for asphalt mix II containing the two antistrip additives.

It should be noted that the calibration curves presented in Figures 2-5 to 2-8 for the two antistrip additives differ because of the different chemical composition of the two additives, different mix gradation and different asphalt types. The R^2 values for these calibration curves are all higher than 0.96. However, the calibration curves are very steep, especially, at additive contents higher than 1%. Therefore, it may be difficult to accurately evaluate higher additive contents in unknown mix samples. The litmus strip saturation problem that was encountered with Morlife 2200 in PG 64-22 Citgo Wilmington asphalt binder was not encountered with the respective mixes, perhaps because the additive adhered more strongly to the aggregate.

Figures 2-9 and 2-10 show the effect of mix type for each antistrip additive, respectively. These comparisons suggest that different mixes (combination of aggregate source and gradation, asphalt binder source and content) require individual calibration curves.

2.4.3 Validation of the litmus test procedure using field samples

To validate the litmus test procedure, field samples of Mix I was obtained from the job site. The hot mix was sampled from the back of the truck just before the lay-down operation and immediately placed into testing cans that were sealed with masking tape. The JMF requires that field mix I contains 0.5% antistrip additive LOF 6500 by asphalt binder weight.

Twenty field samples were analyzed in the laboratory according to the litmus test procedure outlined for mix testing. The color index was obtained and the antistrip additive LOF 6500 content was computed using the regression equation shown in Figure 2-5. That is,

$$C = 1.89 \times 10^{-7} i^3 - 2.70 \times 10^{-4} i^2 + 1.34 \times 10^{-1} i - 22.28 \qquad \text{Eq. (2.1)}$$

where, C is calculated additive content (%), *i* is color index value.
The predicted values are shown in Table 2-12.

The field sample test results show that antistrip additive in mix I ranged from 0.49% to 0.58%, with a mean value of 0.55% and a coefficient of variation of 4.83%. The test results indicate that the litmus procedure is able to qualitatively detect the presence of antistrip additive in the field mix as well as to quantify the amount in the field mix that was tested in this study. However, to confirm these results, further verification is needed using more mixes, different antistrip additives and dosages.

2.4.3 Summary and conclusion

This study presents a relatively simple method for detecting and quantifying amine-based antistrip additives in asphalt binders and mixes. The developed procedure was based on a litmus test that produced results that are quantifiable and repeatable with low coefficients of variation. The conclusions based on the results of this study are:

- The litmus test appears to be capable of detecting and quantifying organic antistrip additives (at least those used in this study).
- For both asphalt binders and mixes, the color index was strongly correlated with antistrip additive content (R^2 > 0.92). The relationships were particularly strong for asphalt mixes, with R^2 values greater than 0.96.
- The relationship between color index and antistrip additive content is unique for each asphalt binder and mix containing a given additive type.
- The litmus test method was capable of correctly determining the amount of antistrip

additive in a field mix.

- The repeatability of the test based on the analysis of 20 replicate field samples had a coefficient of variation of less than 5%. However, these results are based on one field mix, with tests conducted by a single operator. It is anticipated that the coefficient of variation will be higher when testing is conducted by multiple operators in multiple laboratories. Therefore, further verification is needed using more mixes, different antistrip additives and dosages.

Table 2-1 Physical Property of Antistrip Additives

Physical Parameter	LOF 6500	Morlife 2200
Boiling Point	>500ºF	716ºF
Solubility in Water	Slight	Miscible
Vapor Pressure (mmHg at 25ºC)	<1	Not Established
Vapor Density (Air= 1)	>1	Not Established
Appearance	Dark brown liquid	Brown dark liquid
Odor	Mild	Slight
Specific Gravity (at 25ºC)	0.96-0.98	1.06

Table 2-2 Blend Percentages, Material Type I

Material	Blend %
Coarse Aggregate,#78M	15.0
Coarse Aggregate,#67	47.0
Recycled Asphalt Pavement (RAP), Coarse	15.0
Screenings, Regular	16.0
Sand, Natural	7.0
Total	100.0

Table 2-3 Gradation, Material Type I

Sieve Size (mm)	Passing %
50.0	100
37.5	100
25.0	100
19.0	98
12.5	77
9.5	64
4.75	39
2.36	27
1.18	22
0.6	13
0.3	8
0.15	6
0.075	4.1

Table 2-4 Blend Percentages, Material Type II

Material	Blend %
Coarse Aggregate,#57	15.7
Coarse Aggregate,#78M	31.0
Recycled Asphalt Pavement (RAP), Coarse	14.9
Screenings, Asphalt	7.0
Screenings, Concrete	26.2
Sand, Pit	5.2
Total	100.0

Table 2-5 Gradation, Material Type II

Sieve Size (mm)	Passing %
50.0	100
37.5	100
25.0	100
19.0	98
12.5	90
9.5	84
4.75	60
2.36	44
1.18	31
0.6	26
0.3	16
0.15	8
0.075	4.1

Table 2-6 Data for LOF 6500 Additive in PG 64-22 Citgo Wilmington Asphalt Binder, Color Index

Antistrip Additive Content (%)	Color Index														
	Test 1			Test 2			Test 3			Test 4			Test 5		
0	448	429	431	428	434	423	433	434	423	419	425	429	419	437	444
0.25	731	720	717	663	681	675	713	700	692	612	622	620	612	600	592
0.5	728	739	730	787	796	806	702	697	691	769	754	752	860	861	854
1	920	931	926	901	892	890	956	967	966	942	929	940	899	909	887
2	1005	989	995	1020	1040	1021	992	989	990	1002	1009	999	1042	1060	1062

Table 2-7 Data for Morlife 2200 Additive in PG 64-22 Citgo Wilmington Asphalt Binder, Color Index

Antistrip Additive Content (%)	Color Index														
	Test 1			Test 2			Test 3			Test 4			Test 5		
0	448	429	431	428	434	423	433	434	423	419	425	429	419	437	444
0.25	712	707	690	649	630	654	615	595	598	677	668	659	622	631	610
0.5	1017	1027	1012	1000	1019	1026	983	977	969	1001	989	1011	988	976	970
1	1042	1029	1037	1031	1028	1033	1048	1034	1029	1033	1026	1031	1040	1038	1033
2	1008	998	992	995	997	1006	996	998	1000	1003	991	991	997	992	994

Table 2-8 Data for LOF 6500 Additive in Asphalt Mix I, Color Index

Antistrip Additive Content (%)	Color Index					
	Test 1		Test 2		Test 3	
0	388	386	383	384	382	381
0.25	394	401	406	399	404	405
0.5	432	441	440	449	452	458
1	534	542	537	534	535	540
2	612	628	629	610	626	622

Table 2-9 Data for Morlife 2200 Additive in Asphalt Mix I, Color Index

Antistrip Additive Content (%)	Color Index					
	Test 1		Test 2		Test 3	
0	388	386	383	384	382	381
0.25	468	461	474	472	469	448
0.5	502	498	539	542	488	490
1	562	553	543	545	550	533
2	598	600	602	588	537	540

Table 2-10 Data for LOF 6500 Additive in Asphalt Mix II, Color Index

Antistrip Additive Content (%)	Color Index					
	Test 1		Test 2		Test 3	
0	428	423	418	419	422	424
0.25	438	441	444	449	432	430
0.5	474	480	501	510	520	512
1	598	605	627	630	619	617
2	714	715	685	690	739	732

Table 2-11 Data for Morlife 2200 Additive in Asphalt Mix II, Color Index

Antistrip Additive Content (%)	Color Index					
	Test 1		Test 2		Test 3	
0	428	423	418	419	422	424
0.25	433	437	445	440	435	439
0.5	498	495	468	474	482	486
1	604	597	569	576	592	583
2	865	862	854	857	859	860

Table 2-12 Results for LOF 6500 Additive in Mix I, Field Samples

Specimen	Color Index	Additive Content (%)
1	451	0.57
2	446	0.54
3	447	0.55
4	451	0.57
5	437	0.49
6	439	0.50
7	449	0.56
8	453	0.58
9	451	0.57
10	446	0.54
11	448	0.56
12	452	0.58
13	452	0.58
14	449	0.56
15	444	0.53
16	441	0.51
17	450	0.57
18	444	0.53
19	447	0.55
20	445	0.54
Average	447	0.55
Standard Deviation (*s*)	4.45	0.027
Coefficient of Variation (*cv*) (%)	1.00	4.83

Figure 2-1 StripScan Device for Measuring Antistrip Additives in Asphalt Binders and Mixes

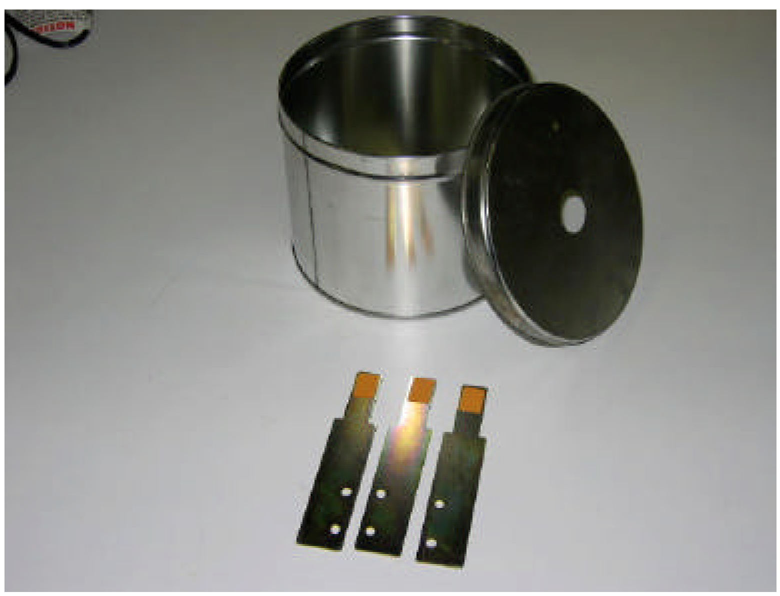


Figure 2-2 Sample Heating Can and Litmus Strips

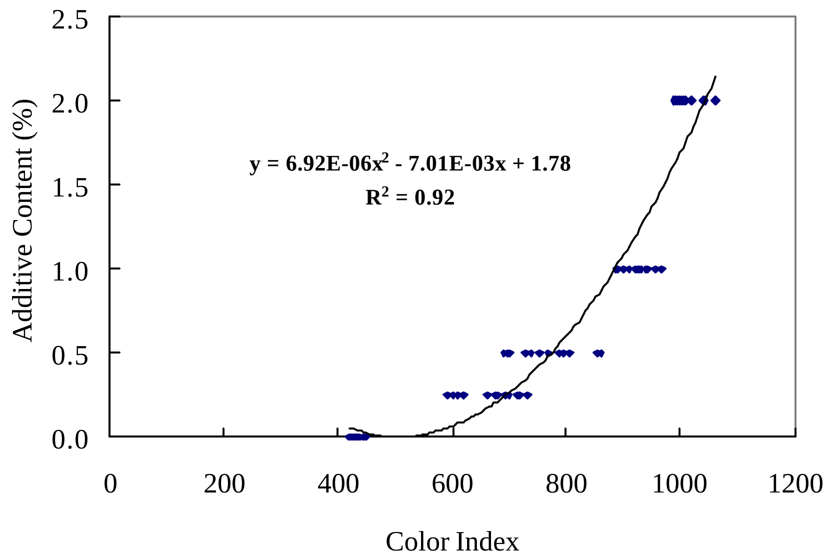


Figure 2-3 Calibration Curve for LOF 6500 Additive in PG 64-22 Citgo Wilmington Asphalt Binder, Percent Additive vs. Color Index

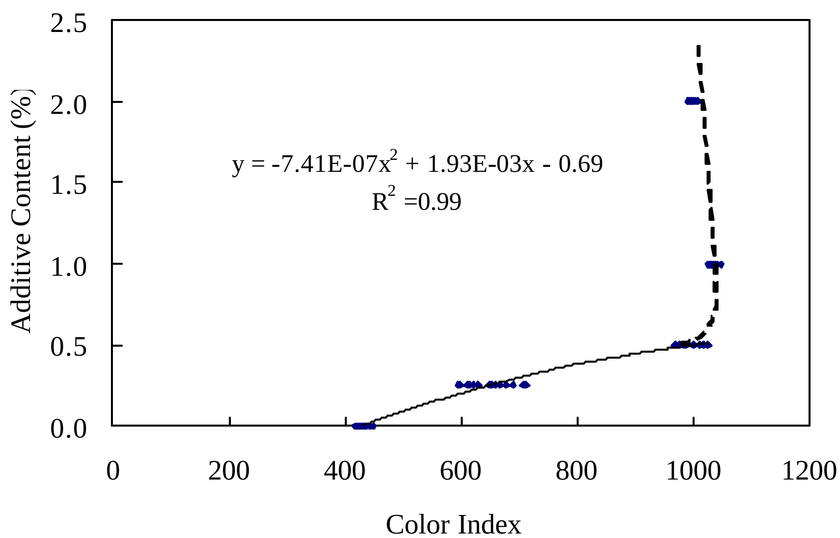


Figure 2-4 Calibration Curve for Morlife 2200 Additive in PG 64-22 Citgo Wilmington Asphalt Binder, Percent Additive vs. Color Index

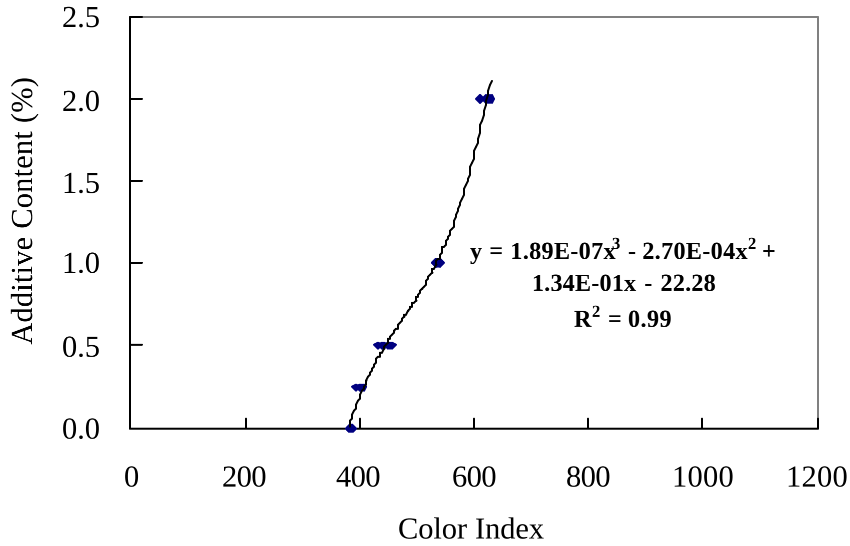


Figure 2-5 Calibration Curve for LOF 6500 Antistrip Additive in Mix I, Percent Additive vs. Color Index

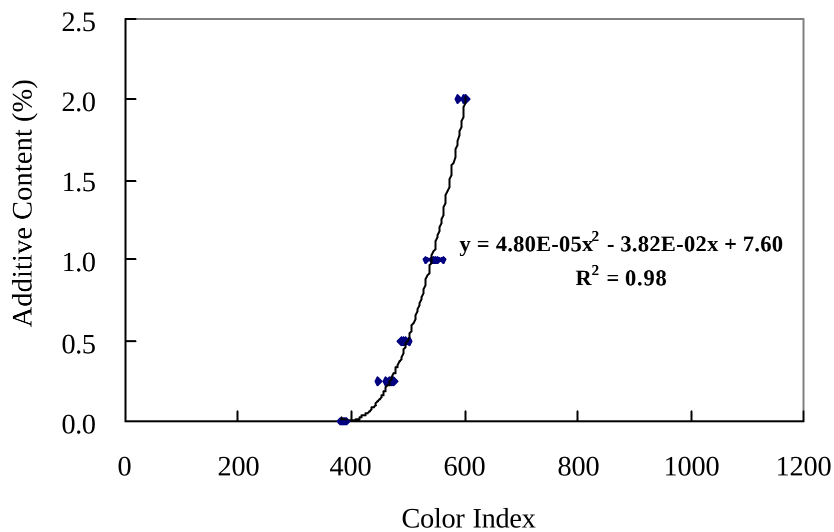


Figure 2-6 Calibration Curve for Morlife 2200 Antistrip Additive in Mix I, Percent Additive vs. Color Index

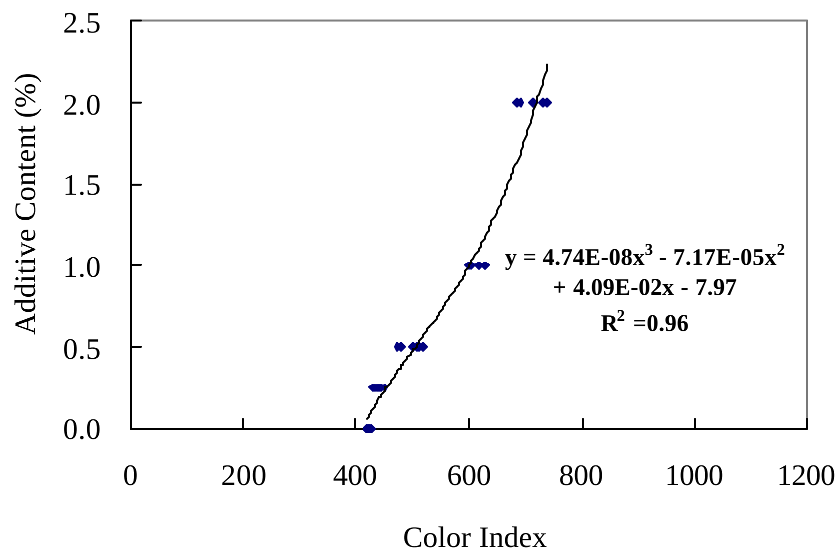


Figure 2-7 Calibration Curve for LOF 6500 Antistrip Additive in Mix II, Percent Additive vs. Color Index

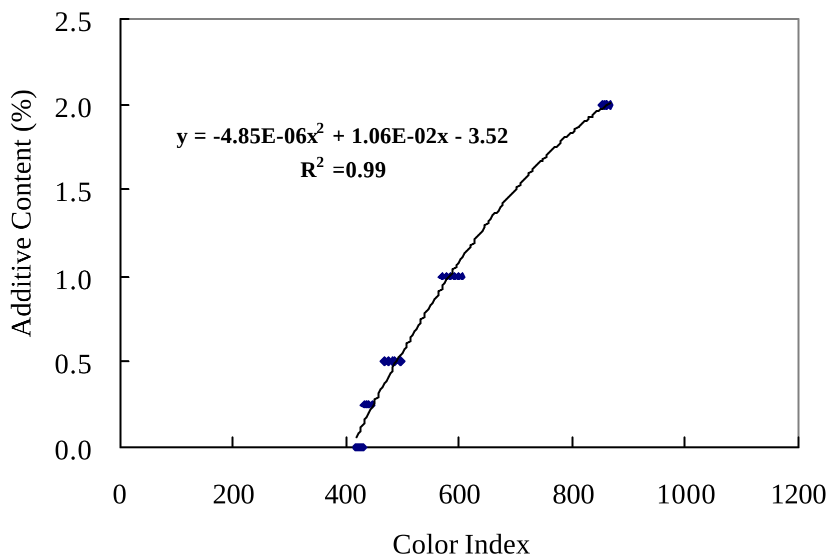


Figure 2-8 Calibration Curve for Morlife 2200 Antistrip Additive in Mix II, Percent Additive vs. Color Index

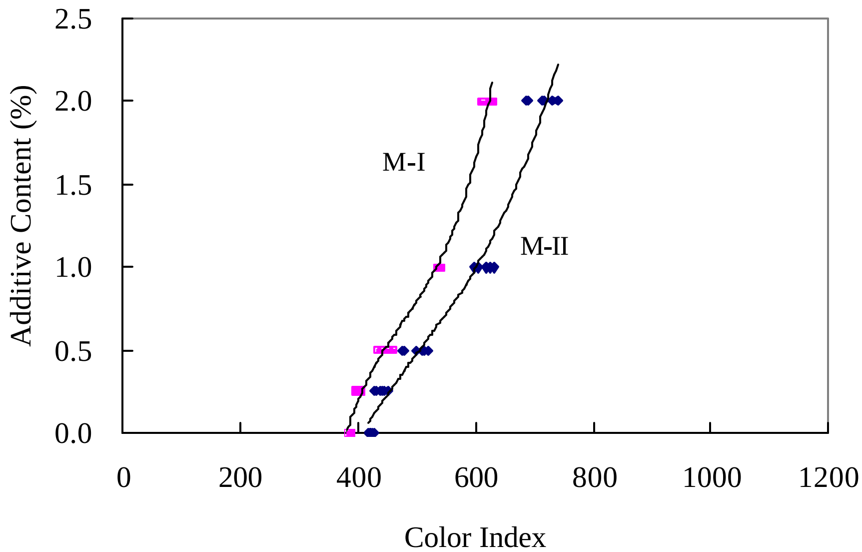


Figure 2-9 Comparison of Calibration Curves for Mix I and Mix II Containing LOF 6500 Antistrip Additive

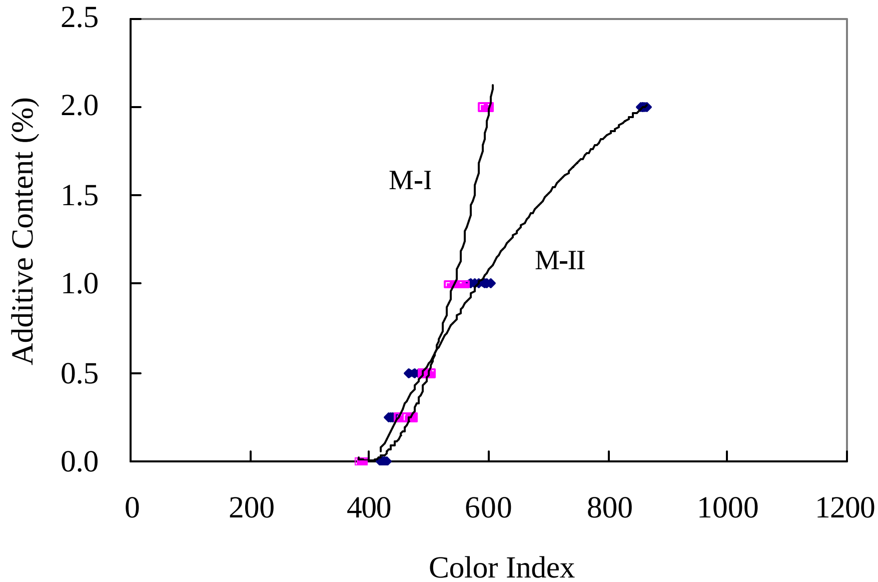


Figure 2-10 Comparison of Calibration Curves for Mix I and Mix II Containing Morlife 2200 Antistrip Additive

3. Quantifying Organic Antistrip Additive Using Colorimetric Test Method

3.1 Colorimetric Test Overview

Various methods for the spectrophotometric determination of amines in aqueous solution have been developed since the 1960s [13-15]. Silverstein [14] established a spectrophotometric method for quantifying the aqueous concentrations of fatty amines. To determine the total amine concentration in water, methyl orange was allowed to react with the amines in the presence of an acetate buffer, and the resulting yellow complex was extracted into ethylene dichloride. The method was effective for amine analyses in the parts per million (ppm) concentration range. Larrick [15] improved on Silverstein's spectrophotometric analysis method by combining the buffer and dye reagents into a combination reagent. In this study, the aqueous solution of amines was produced by dissolving volatiles emitted during heating of asphalt binders and mixes in a water trap. Details of the procedure and the device for trapping amine vapors into solution are outlined below.

The colorimetric test for quantifying amine-based antistrip additives in asphalt binders and mixes was developed as a three-step process consisting of: (1) amine extraction and trapping, (2) chemical reaction with color reagent, and (3) absorbance measurement. First, during the extraction and trapping process, a preheated sample of asphalt binder or mix was transferred to a hot plate and maintained at a temperature of 170°C. For a period of 10 minutes, emitted gases were transported with the N_2 carrier gas to a gas washing bottle containing 100 mL of ultra pure water. The time measurement was started when air bubbles first appeared in the water. Second, in the chemical reaction process, 5 mL of Larrick's combined reagent [15] was added to 100 mL deionized water taken from the gas washing bottle and allowed to react for a period of 10 min. Following the addition of 20 mL of ethylene dichloride, the colored complex formed in step 2 was extracted into the organic solvent by shaking the sample for 5 min. Upon phase separation, an ethylene dichloride sample was removed, and the absorbance of the ethylene dichloride extract was measured in a 1-cm path length glass cuvette at a wavelength of 420 nm using a spectrophotometer (UV1, Spectronic Unicam, England). An ethylene dichloride extract obtained using asphalt binder or mix without antistrip additive was used to establish a baseline reading for absorbance measurements. The absorbance values of the ethylene dichloride extracts obtained from samples containing different levels of antistrip additive contents were compared with the baseline absorbance value, and the difference between the sample and baseline values was used to quantify the amount of antistrip additive in asphalt binders or mixes.

3.2 Device for Extracting Antistrip Additives from Asphalt Binders and Mixes

The colorimetric technique requires that amines are present in aqueous solution. Therefore, an amine trapping system was developed during the course of this study. A schematic representation is shown in Figure 3-1. This device consisted of a nitrogen gas cylinder, valve, tube, heating tape, flow meter, sample container, heater, and a gas washing bottle containing deionized water. The functions of individual components are stated in detail as follows:

a. Nitrogen tank: Inert N_2 gas under pressure was used to facilitate the transport of volatiles from the sample container into the gas washing bottle (Fisher Scientific, Hampton, NH) containing deionized water.
b. Valve: Start or stop N_2 gas flow from the gas tank.
c. Stainless steel tube: Transfer N_2 gas from tank to sample container and deionized water.
d. Heating tape: Heat N_2 gas flowing in stainless steel tubes to avoid condensation of asphalt fumes.
e. Flow meter: N_2 gas flow rate was controlled by the flow meter at 50 cm^3/min. (shown in Figure 3-2)
f. Sample container: Two sizes of sealed metal cans were used to heat samples: A

quarter-gallon can was used for asphalt binders, and a gallon-can was used for mixes (Figures 3-3 and 3-4 show asphalt binder and asphalt mix sample containers, respectively). For analyses, 100-g asphalt binder and 2000-g asphalt mix samples were used. The operator needs to assure that the sample container is fully sealed when testing, otherwise, air leakage will prevent transport of the emitted gas from the sample container to the water trap.

g. Heater: The test sample temperature was maintained at 170°C during the extraction process.

h. Gas washing bottle: A gas washing bottle containing deionized water was used to transfer amines from the gaseous to the aqueous phase.

3.3 Procedure to Quantify Antistrip Additives in Asphalt Binders and Mixes

Using the above stated components, the following test conditions were used to extract antistrip additives from asphalt binders and mixes: (1) samples were maintained at a temperature of 170ºC, (2) the N_2 gas flow rate was 50 cm^3/min, and (3) the extraction time (time that N_2 flowed through the extraction system) was 10 min.

The preparation procedure of asphalt binder and asphalt mix samples for colorimetric test is similar to the litmus test outlined previously.

3.3.1 Calibration procedure to determine antistrip additive content in asphalt binder

Based on the experience obtained with the litmus test, the following experimental procedure was developed and followed for the colorimetric method:

1. Based on the desired quantity of asphalt binder, weigh out the required mass of antistrip additive and place into a large metal can. For example, to obtain a 0.25% additive content in 300-g of asphalt binder, 1.25-g additive is weighed out.

Subsequently, pour 300-g of heated asphalt into the can containing the measured additive content.

2. Mix thoroughly for at least 3 minutes to ensure that the additive is uniformly distributed in the asphalt binder. A low-shear mixer of the type used for producing modified asphalt binders may be used.
3. Immediately after mixing, pour 100-g samples into individual test cans and close the can lid. In this study, quarter gallon can size was used as shown in Figure 3-3. The individual cans containing 100-g of asphalt binder with additive may be stored temporarily at room temperature.
4. Using steps 1 to 3, prepare samples with different levels of antistrip additive contents. In this study, five levels of antistrip additive content were evaluated? 0, 0.25, 0.5, 1.0 and 2.0% by weight of asphalt binder.
5. Prior to testing, heat the sample in a forced draft oven at 170°C for 15 minutes. It should be noted that for multiple sample testing, the samples should not all be heated simultaneously. The sample heating process should be staggered based on operator efficiency such that while a given sample is being analyzed with the colorimetric test, the other samples are not being over conditioned by heating them for more than 15 minutes.
6. Prior to testing, prepare 500 mL color reagent (see Figure 3-5a). The procedure for preparing color reagent is as follows: Dissolve 0.1 g of methyl orange in 100 mL deionized water; dissolve 29.6-g of sodium acetate trihydrate and 50-g of potassium chloride in separate 100 mL deionized water. Combine these two 100 mL solutions and add 100 mL of glacial acetic acid to it. Finally dilute this 300 mL solution to 500 mL with deionized water. [15].
7. Immediately prior to starting the extraction procedure, turn on the heater and heating tape such that the temperature of the sample and the vapor transfer lines are maintained at 170°C, then open the valves of the N_2 gas tank.
8. Transfer the heated sample to the amine trapping device and allow the vapors to pass through 100 mL deionized water for 10 min. A stopwatch is started when air bubbles begin to appear in the water of the gas washing bottle. Nitrogen gas at a flow rate of 50 cm^3/min is used to facilitate the transfer of vapors from the can

containing the asphalt sample to the wash bottle containing deionized water.

9. Add 5 mL of the color reagent to the acidified water in the wash bottle (from step 8) and set aside for 10 min (see Figure 3-5b). Add 20 mL of ethylene dichloride and shake for 5 min. Allow the layers to separate for 5 min (see Figure 3-5c) [15].
10. Extract 1-2 mL of the ethylene dichloride phase (lower layer) and place into a glass vial (see Figure 3-5d).
11. Repeat the procedure steps 5, 8, 9, 10 for samples containing different levels of antistrip additive contents.
12. Measure absorbance of the ethylene dichloride sample immediately at 420 nm using a spectrophotometer. The ethylene dichloride extract obtained from an asphalt sample without added antistrip additive is used as the baseline absorbance value measured with the spectrophotometer (UV1, Spectronic Unicam, England, shown in Figure 3-6), that is, use this extract to zero the spectrophotometer.
13. Establish a calibration curve (regression equation) between the additive content and the absorbance values determined by the spectrophotometer.

3.3.2 Calibration procedure to determine antistrip additive content in asphalt mixes

The preparation procedure of asphalt mix sample is the same as that for litmus test and the colorimetric test procedure is similar to that described for asphalt binder.

1. Incorporate the required amount of antistrip in asphalt binder. This process is the same as that presented above.
2. Prepare asphalt mix samples containing different antistrip additive contents. In this study, three 2000-g mix samples were prepared for each level of antistrip additive content. Note that the sample container needs to be replaced with a larger 1 gal. can, as shown in Figure 3-4.
3. Before testing, preheat the sample for 1 hour at 170°C. Leave the lid open when heating and stir (agitate) the sample every 15-30 minutes.
4. Follow steps 8 to 13 outlined above for the asphalt binder.

3.4 Test Results and Discussion

3.4.1 Test results for antistrip additive in asphalt binder

For the colorimetric test, two antistrip additives (LOF 6500 and Morlife 2200) were added to PG 64-22 Citgo Wilmington asphalt binder at levels of 0, 0.25, 0.5, 1.0 and 2.0% as previously described. Antistrip additive was heat-extracted from the asphalt binder and trapped in water. Following reaction with a color reagent, the aqueous reaction product was extracted into a small volume of organic solvent and the color intensity (absorbance) of the solvent extract was quantified by spectrophotometry. The absorbance of the ethylene dichloride extract from an asphalt binder sample without antistrip additive was used as the baseline.

The raw absorbance data obtained for asphalt binder samples, prepared in triplicate at each of five tested additive levels, are shown in Tables 3-1 and 3-2 for LOF 6500 and Morlife 2200, respectively. Calibration curves for the two antistrip additives in PG 64-22 Citgo Wilmington asphalt binders were developed from the data of Tables 3-1 and 3-2 and are shown in Figures 3-7 and 3-8 for LOF 6500 and Morlife 2200, respectively. Figures 3-7 and 3-8 show the relationship between absorbance and antistrip additive content is approximately linear ($R^2 > 0.95$). It is reasonable that the concentration of colored antistrip additive reaction product extracted into ethylene dichloride is proportional to the amount of antistrip additive in asphalt binder. The steeper slope obtained with the Morlife 2200 antistrip additive suggests that Morlife 2200 is either evaporated more easily from asphalt binder or more reactive with the color reagent than LOF 6500. As a consequence, different types of antistrip additive have unique chemical properties and require the development of individual calibration curves. Overall, Figures 3-7 and 3-8 illustrate that the colorimetric test method is effective in detecting not only the presence of antistrip additive but also the amount of antistrip additive in asphalt binder.

3.4.2 Test results for antistrip additives in asphalt mixes

In this study testing was conducted on mixes I and II containing both LOF 6500 and Morlife 2200 antistrip additive.

The two types of asphalt mix samples were prepared using LOF 6500 and Morlife 2200 antistrip additives at levels of 0, 0.25, 0.5, 1.0 and 2.0% by asphalt binder weight. For each level of antistrip additive content, three replicate samples, each weighing 2000-g, were prepared. Tables 3-3 and 3-4 show the absorbance data obtained at 420 nm wavelength for asphalt mix I containing the two antistrip additives LOF 6500 and Morlife 2200, respectively. Similarly, Tables 3-5 and 3-6 show the absorbance data obtained for asphalt mix II containing the two antistrip additives LOF 6500 and Morlife 2200, respectively.

It should be noted that the calibration curves presented in Figures 3-9 to 3-12 for the two antistrip additives differ because of the different chemical composition of the two antistrip additives, different mix gradation and different asphalt types. The R^2 values for these calibration curves are all higher than 0.94. Unlike the linear calibration equations of antistrip additive in asphalt binder, the calibration regression equations of antistrip additive in asphalt mix are quadratic. In addition, these figures show that the calibration equations derived by data of antistrip additive in asphalt mix using colorimetric test are not very accurate for samples containing antistrip additive contents of 0.25% or below.

Figures 3-13 and 3-14 show the effect of mix type for each antistrip additive, respectively. As was the case with litmus testing, these comparisons suggest that different mixes (combination of aggregate source and gradation, asphalt binder source and content) require individual calibration curves.

3.4.3 Validation of the colorimetric test procedure using field samples

As with the litmus test, field samples corresponding to mix I were obtained from the job site to validate the colorimetric test procedure. The hot mix was sampled from the back of the truck just before the lay-down operation and immediately placed into testing cans that were sealed with masking tape. The JMF required field mix I to contain 0.5% LOF 6500 antistrip additive by asphalt binder weight.

Ten field samples were tested in the laboratory according to the colorimetric test procedure outlined for mix testing. The absorbance at 420 nm was obtained and the LOF 6500 antistrip additive content was computed using the regression equation shown in Figure 3-10. That is,

$$C=751.9681a^2+12.0664a+0.0928 \qquad \text{Eq. (3-1)}$$

where, C is calculated additive content (%), *a* is absorbance value.
The predicted values are shown in Table 3-7.

The field sample tests results show that antistrip additive in mix I ranged from 0.38% to 0.66%, with a mean value of 0.49% and a coefficient of variation of 18%. The test results indicate that the colorimetric procedure is able to qualitatively detect the presence of antistrip additive in the field mix as well as to quantify the amount.

3.4.4 Summary and Conclusion

In this study, an amine trapping system was developed and used to extract organic antistrip additive from asphalt binders and mixes into aqueous solution. The developed procedure was based on a colorimetric test that produced results that are quantifiable and repeatable. The conclusions based on the results of this study are:

- The designed extraction device can successfully extract organic antistrip additive from asphalt binders and mixes into deionized water, which makes possible the detection of antistrip additive in asphalt by a colorimetric method.

- For both asphalt binders and mixes, the absorbance at 420 nm was strongly correlated with antistrip additive content ($R^2 > 0.94$).
- The colorimetric test is capable of detecting and quantifying organic antistrip additive (at least those used in this study).
- The relationship between absorbance and antistrip additive content is unique for each asphalt binder and mix containing a given additive type.
- The colorimetric test method was capable of determining the amount of antistrip additive in a field mix.
- The repeatability of the test based on the analysis of 10 replicate field samples was acceptable with a coefficient of variation of approximately 18%.

Table 3-1 Data for LOF 6500 Additive in PG 64-22 Citgo Wilmington Asphalt Binder, Absorbance

Antistrip Additive Content (%)	Absorbance		
	Test 1	Test 2	Test 3
0	0	0	0
0.25	0.0030	0.0022	0.0018
0.5	0.0103	0.0089	0.0078
1	0.0124	0.0146	0.0174
2	0.0329	0.0297	0.0273

Table 3-2 Data for Morlife 2200 Additive in PG 64-22 Citgo Wilmington Asphalt Binder, Absorbance

Antistrip Additive Content (%)	Absorbance		
	Test 1	Test 2	Test 3
0	0	0	0
0.25	0.0018	0.0032	0.0041
0.5	0.0122	0.0096	0.0137
1	0.0287	0.0252	0.0273
2	0.0405	0.0422	0.0373

Table 3-3 Data for LOF 6500 Additive in Asphalt Mix I, Absorbance

Antistrip Additive Content (%)	Absorbance		
	Test 1	Test 2	Test 3
0	0	0	0
0.25	0.0030	0.0038	0.0024
0.5	0.0145	0.0206	0.0171
1	0.0285	0.0257	0.0290
2	0.0410	0.0429	0.0441

Table 3-4 Data for Morlife 2200 Additive in Asphalt Mix I, Absorbance

Antistrip Additive Content (%)	Absorbance		
	Test 1	Test 2	Test 3
0	0	0	0
0.25	0.0054	0.0067	0.0062
0.5	0.0282	0.0257	0.0245
1	0.0401	0.0393	0.0457
2	0.0509	0.0556	0.0602

Table 3-5 Data for LOF 6500 Additive in Asphalt Mix II, Absorbance

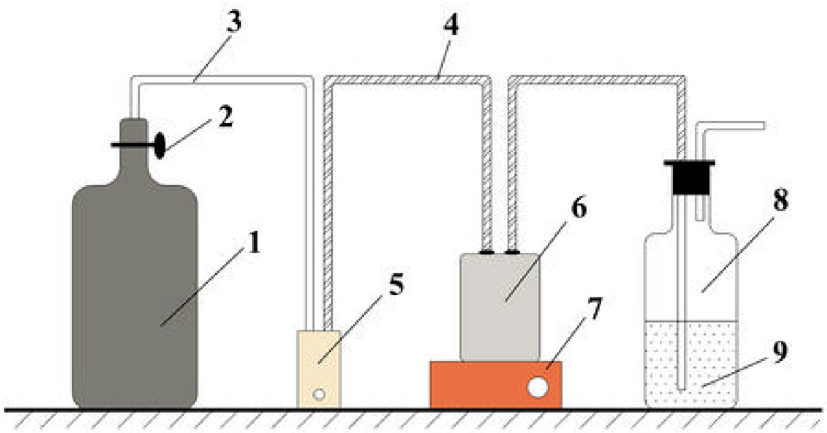
Antistrip Additive Content (%)	Absorbance		
	Test 1	Test 2	Test 3
0	0	0	0
0.25	0.0027	0.0024	0.0019
0.5	0.0223	0.0247	0.0212
1	0.0363	0.0380	0.0404
2	0.0542	0.0510	0.0483

Table 3-6 Data for Morlife 2200 Additive in Asphalt Mix II, Absorbance

Antistrip Additive Content (%)	Absorbance		
	Test 1	Test 2	Test 3
0	0	0	0
0.25	0.0043	0.0052	0.0029
0.5	0.0379	0.0357	0.0342
1	0.0569	0.0579	0.0552
2	0.0789	0.0756	0.0744

Table 3-7 Results for LOF 6500 Additive in Mix I, Field Samples

Specimen NO.	Absorbance	Additive Content (%)
1	0.0154	0.46
2	0.0192	0.60
3	0.0137	0.40
4	0.0178	0.55
5	0.0165	0.50
6	0.0142	0.42
7	0.0171	0.52
8	0.0206	0.66
9	0.0131	0.38
10	0.0151	0.45
Average	0.0163	0.49
Standard Deviation (s)	0.0024	0.09
Coefficient of Variation (cv) (%)	14.9827	18.47



1. Nitrogen tank
2. Valve
3. Tubing
4. Heating Tape
5. Flow meter
6. Sample container
7. Heater
8. Gas washing bottle
9. Deionized water

Figure 3-1 Schematic of Amine Trapping System

Figure 3-2 Flow Meter used to Control N_2 Gas Flow Rate

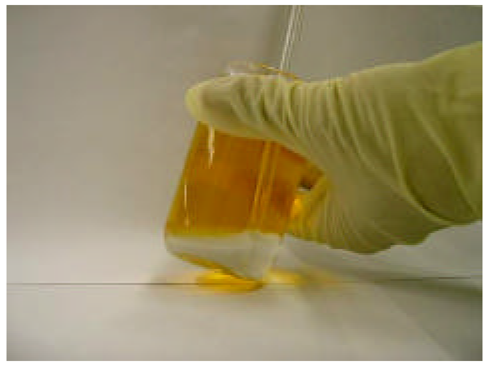
Figure 3-3 Extraction Device for Transferring Antistrip Additive from Asphalt Binder to the Aqueous Phase

Figure 3-4 Extraction Device for Transferring Antistrip Additive from Asphalt Mix to the Aqueous Phase

a) Preparation of color reagent

b) Add color reagent to acidified water and set aside

c) Phase separation



d) Extraction of ethylene dichloride phase

Figure 3-5 Chemical Reaction of Aqueous Extract with Dye and Extraction of Solvent

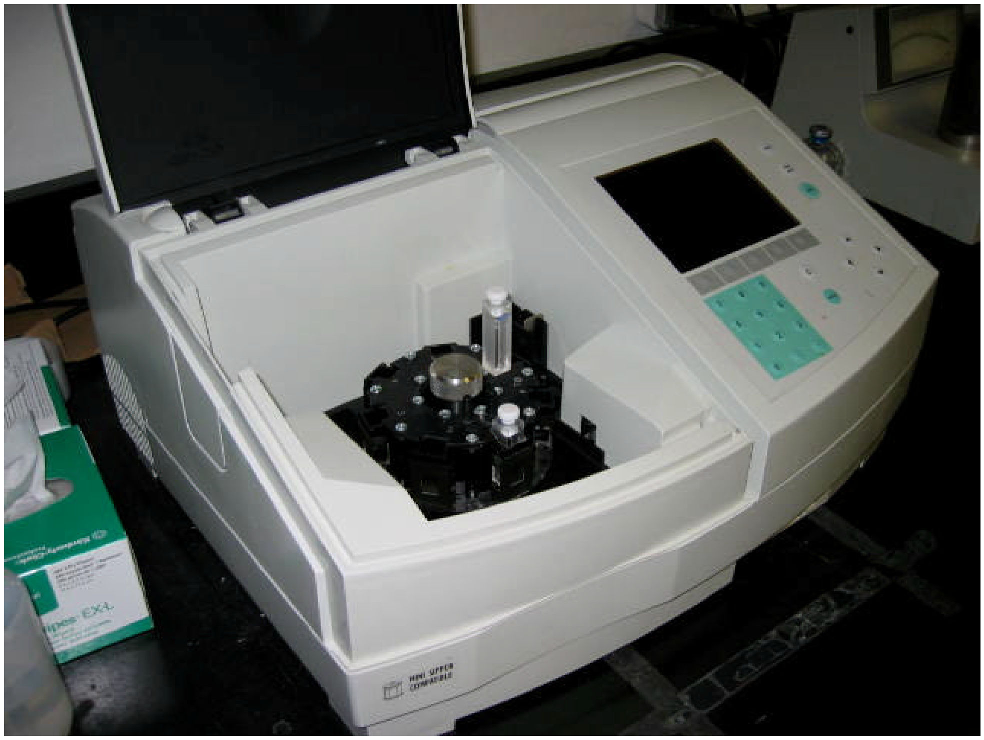


Figure 3-6 Spectrometer for Measurement of Absorbance of Ethylene Dichloride Extracts

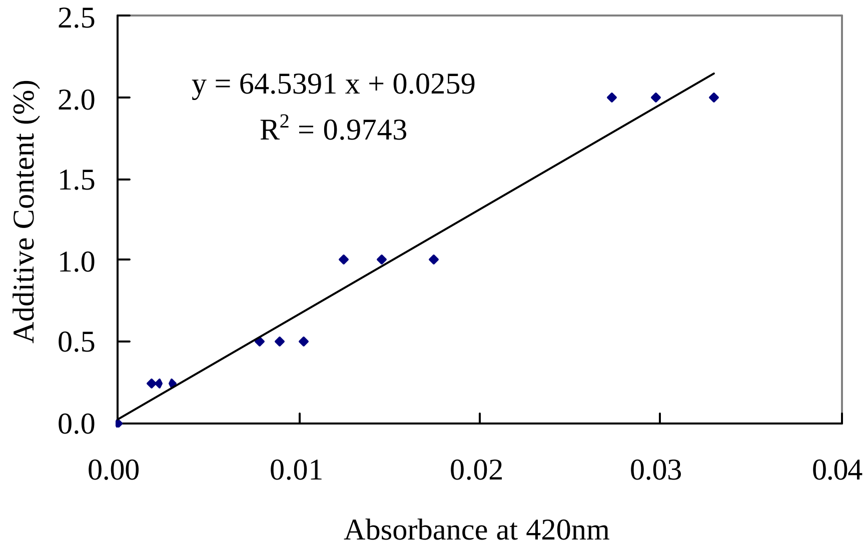


Figure 3-7 Calibration Curve for LOF 6500 Additive in PG 64-22 Citgo Wilmington Asphalt Binder, Percent Additive vs. Absorbance

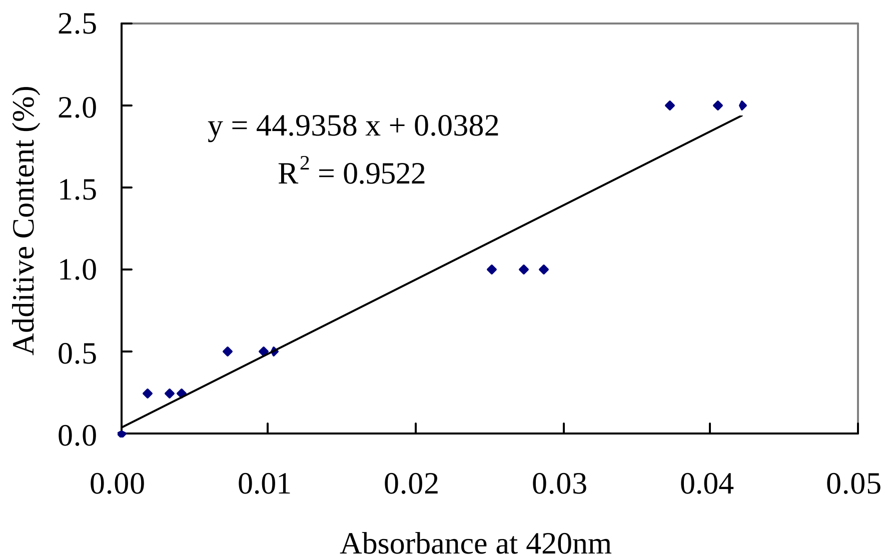


Figure 3-8 Calibration Curve for Morlife 2200 Additive in PG 64-22 Citgo Wilmington Asphalt Binder, Percent Additive vs. Absorbance

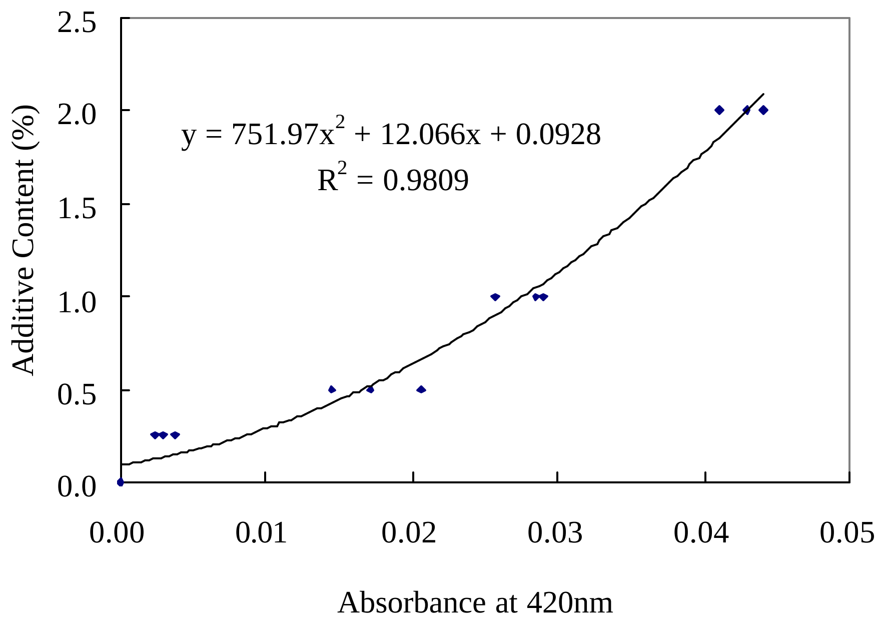


Figure 3-9 Calibration Curve for LOF 6500 Antistrip Additive in Mix I, Percent Additive vs. Absorbance

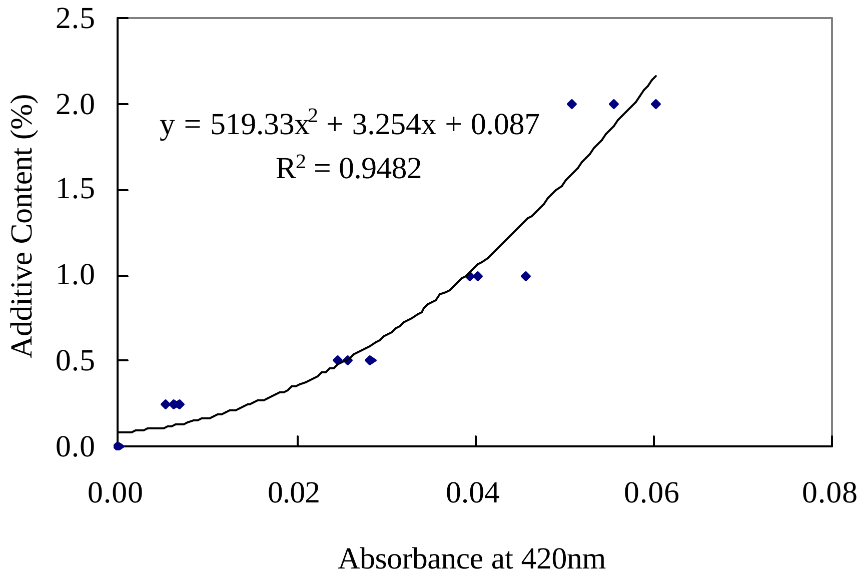


Figure 3-10 Calibration Curve for Morlife 2200 Antistrip Additive in Mix I, Percent Additive vs. Absorbance

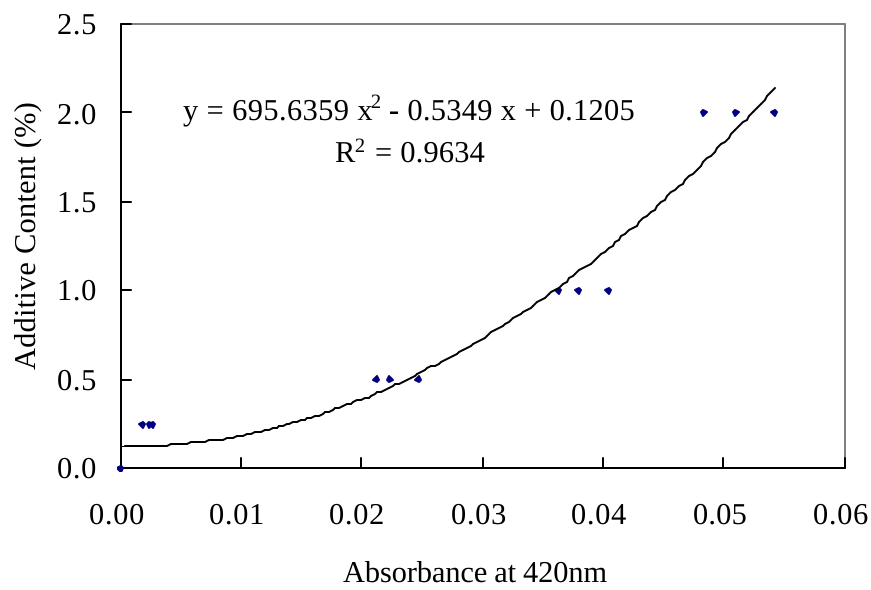


Figure 3-11 Calibration Curve for LOF 6500 Antistrip Additive in Mix II, Percent Additive vs. Absorbance

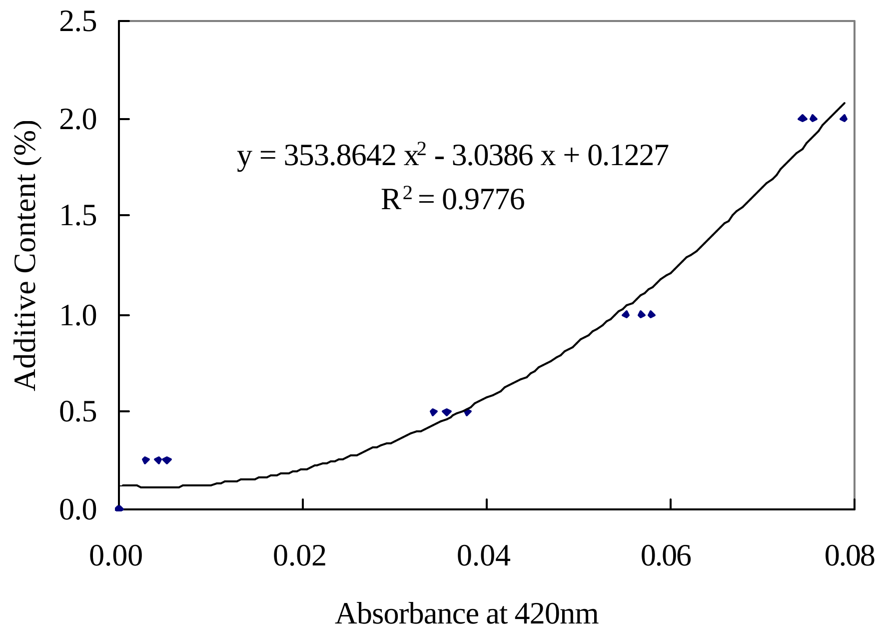


Figure 3-12 Calibration Curve for Morlife 2200 Antistrip Additive in Mix II, Percent Additive vs. Absorbance

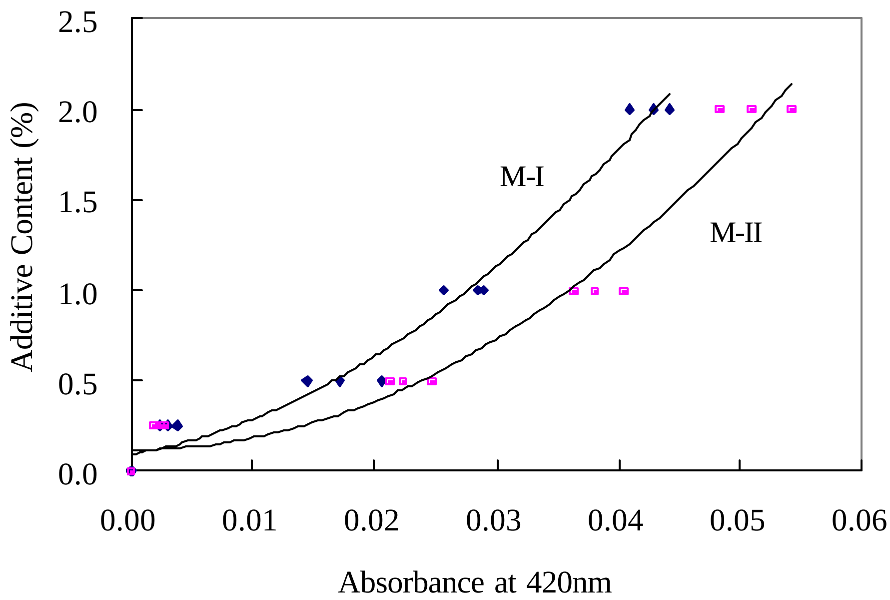


Figure 3-13 Comparison of Calibration Curves for Mix I and Mix II Containing LOF 6500 Antistrip Additive

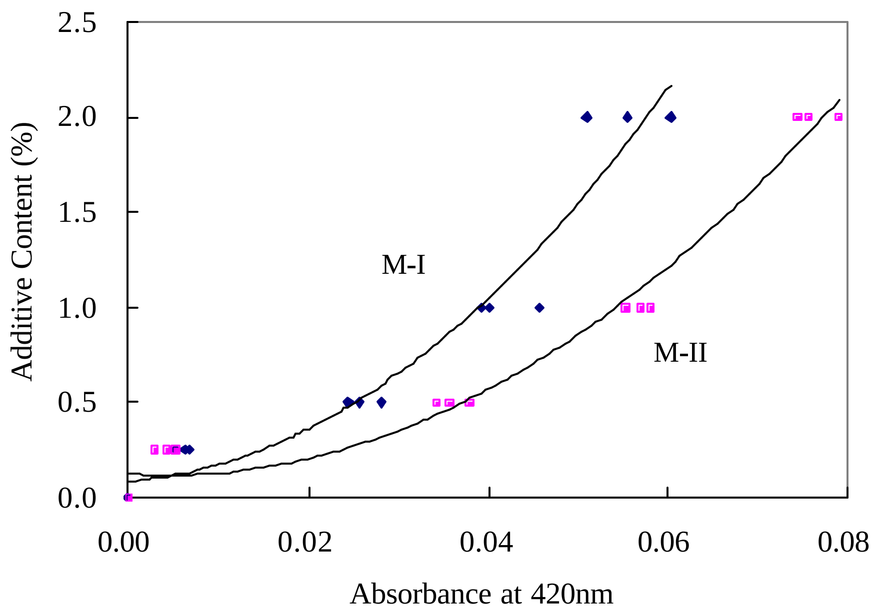


Figure 3-14 Comparison of Calibration Curves for Mix I and Mix II Containing Morlife 2200 Antistrip Additive

4. Effect of Prolonged Heating on Antistrip Additive Contents in Asphalt Binders and Mixes

4.1 Introduction

The loss of amine-based antistrip additives from asphalt binders and mixes through volatilization has long been suspected in the asphalt industry. Due to this reason, many highway agencies now require the use of lime as antistrip additive in mixes. The use of litmus and colorimetric tests offers a viable method to study the effect of prolonged heating on the loss of amine-based antistrip additives in both asphalt binders and mixes. The effect of prolonged heating on the loss of antistrip additives is explored in the following section.

4.2 Thermal Analysis of Pure Antistrip Additives

Both antistrip additives LOF 6500 and Morlife 2200 were subjected to thermal analysis to evaluate the effect of prolonged heating on the mass loss. Figure 4-1 shows the mass loss in percent as a function of time at 150°C temperature for both antistrip additives. Based on Figure 4-1, Morlife 2200 antistrip additive is more volatile as compared to LOF 6500 antistrip additive. The mass loss is rapid in the first 6 to 8 hours of heating and tapers off after about 24 hours of heating. In 48 hours, the mass loss for antistrip additives LOF 6500 and Morlife 2200 is roughly 30 and 50 percent, respectively.

Based on Figure 4-1, it is apparent that the higher molecular weight fraction in both antistrip additives does not volatilize easily. However, visual inspection of the antistrip additive LOF 6500 sample shown in Figure 4-2 indicates a distinct color change as function of heating time. After approximately 12 hours of heating, the clear liquid turns to black sticky mass probably indicating decomposition of the non-volatile fraction.

The sticky mass was observed to solidify upon cooling. Similar observations were also made for the Morlife 2200 antistrip additive.

4.3 Changes in Antistrip Additive Contents in Asphalt Binders and Mixes after Prolonged Heating

In order to explore the loss of additive due to prolonged heating, the litmus and colorimetric test procedures presented in Chapters 2 and 3 were repeated for asphalt binder samples containing different levels of antistrip additive after 2, 6, 24, and 48 hours of prolonged heating. For asphalt mix samples containing different levels of antistrip additive, tests were repeated after 1, 2, 4, 6, and 12 hours of prolonged heating.

4.3.1 Change of measured antistrip additive content in asphalt binders and mixes after prolonged heating using litmus test

To measure the change in antistrip additive content in asphalt binders and mixes after prolonged heating, the litmus test procedure was used on samples obtained after prolonged heating (2, 6, 24, and 48 hours). Litmus test data are presented in Appendix A. Based on the color index obtained, additive contents were computed from the regression equation presented in Figure 2-3 shown in the chapter 2. Figures 4-3 and 4-4 show the trend in the data graphically for asphalt binder containing LOF 6500 additive.

An important observation from the data obtained can be seen in Figure 4-4 that represents the decline of additive content as a function of prolonged heating. The data shows that after 24 to 48 hours of heating, there is practically no additive left in the asphalt binder. This poses a serious question regarding the effectiveness of the antistrip additives in field mixes – especially, mixes prepared using terminally blended asphalt binders that may be stored at high temperatures for up to 24 hours and sometimes even longer.

Figure 4-5 shows the data for the antistrip additive Morlife 2200. It should be noted from this figure and as discussed previously in chapter 2, that the StripScan is not able to quantitatively detect very high percentages of the Morlife 2200 antistrip additive (beyond 0.5 to 1.0 percent) due to the over saturation of the litmus strip in the StripScan. For this reason Table A-4 in appendix A shows no data entries related to the samples containing 1 and 2% Morlife 2200 antistrip additive. However, up to 0.5-percent, the antistrip additive could be measured accurately. Similar to the LOF 6500 antistrip additive, Figure 4-5 also shows that after extended heating the Morlife 2200 is also evaporating from the asphalt binder. After 24 to 48 hours of extended heating, there is practically no antistrip additive left in the binder.

Figures 4-6 through 4-9 show the effect of prolonged heating on antistrip additive contents in Mix I for LOF 6500 and Morlife 2200 additives. The trend for the loss of antistrip additive (both LOF 6500 and Morlife 2200) in asphalt Mix I is very similar to that obtained for the asphalt binder. A notable difference, however, is that the measured additive content in asphalt mix decreases rapidly in the first 6 hours and the additive content approaches zero in a much shorter time frame than that observed for asphalt binder.

Similar to Mix I, Figures 4-10 through 4-13 show the effect of prolonged heating on antistrip additive contents for Mix II. The observations are the same as for Mix I, that is, after 6 hours of prolonged heating, practically no antistrip additive could be detected.

4.3.2 Change of measured antistrip additive content in asphalt binders and mixes after prolonged heating process using colorimetric test

In order to validate the ability of the litmus test to evaluate the effect of prolonged heating on antistrip additive contents in asphalt binders and mixes, the tests conducted using the litmus test were repeated using the colorimetric analysis.

The colorimetric analysis data are presented in Appendix B. The results are summarized in Figures 4-14 and 4-15; and Figures 4-16 and 4-17 for LOF 6500 and Morlife 2200 additives, respectively. The results of colorimetric analysis validate the results obtained using the litmus test. That is, upon prolonged heating both LOF 6500 and Morlife 2200 antistrip additive contents approach zero after 24 to 48 hours.

Figures 4-18 to 4-25 shows the results for antistrip additive loss in Mixes I and II. The mix test results also validate the litmus test observations of drastic decline in antistrip additive contents in 6 to 12 hours of prolonged heating at nominal storage and compaction temperatures.

4.4 Discussion

The test results presented above raise some interesting questions. First, it is not unreasonable to assume that in many instances, especially for terminally blended asphalt binders, that the binders will be subjected to elevated temperatures for 24 to 48 hours during storage and transport. Second, it is also not unreasonable that mixes in many instances will be subjected to elevated temperatures for 6 to 12 hours during storage and transport. The question is how does the loss of amine-based antistrip additive through volatilization affect the mix performance? Are the DOTs getting more moisture-susceptible mixes and does it translate into inferior pavement performance?

The question of terminally blended asphalt binders is easier to tackle. Based on the results of this study, loss of antistrip additive content is expected by the time terminally blended binder is used at the plant for mix production. The magnitude of the loss will depend on the temperature at which the binder is stored and transported as well as the duration between production and its ultimate use. It is also possible to blend the virgin binder with antistrip additive at the plant just before use, which is preferable but raises the question of antistrip additive segregation. During the course of this study, some issues with segregation were noted; however, this issue was not pursued further and was bypassed by blending smaller batches of binder and immediately pouring the binder into cans using 100-g samples.

In terms of mixes, assuming that a correct dosage of antistrip additive is contained in the mix, does the loss of additive content due to storage and transport at elevated temperatures affect mix performance in terms of moisture sensitivity? This question poses a serious conundrum. Without further research, it is not possible to conclude that the mix will be inferior and more moisture-sensitive. The antistrip additive supposedly facilitates the stronger bond between the asphalt binder and the aggregate. It can be argued that once the antistrip additive has facilitated the bonding process during mixing, volatilization of any excess antistrip additive in the mix may not be of any consequence. Therefore, further research is needed to investigate the effect of extended heating on the bond strength between asphalt and aggregate in relation to loss of antistrip additive content.

4.5 Conclusion

Based on the litmus test and colorimetric analysis, it is evident that there is a loss of antistrip additive content in both asphalt binders and mixes when the asphalt binder or mix is subjected to prolonged heating. However, based on the results of this study, it is neither clear nor possible at this time to conclude what effect if any, does the loss of antistrip additive have on the moisture sensitivity of mixes if the loss occurs after mix production assuming that the specified dosage of antistrip was used during production.

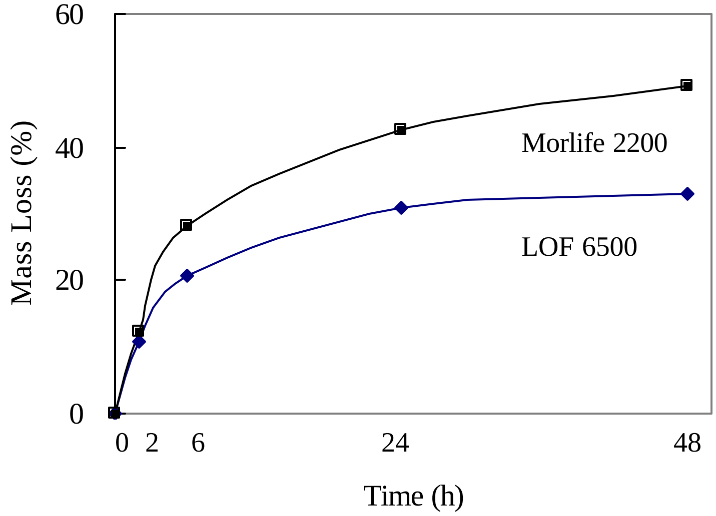


Figure 4-1 Mass Loss of Pure Antistrip Additives as Function of Heating Time

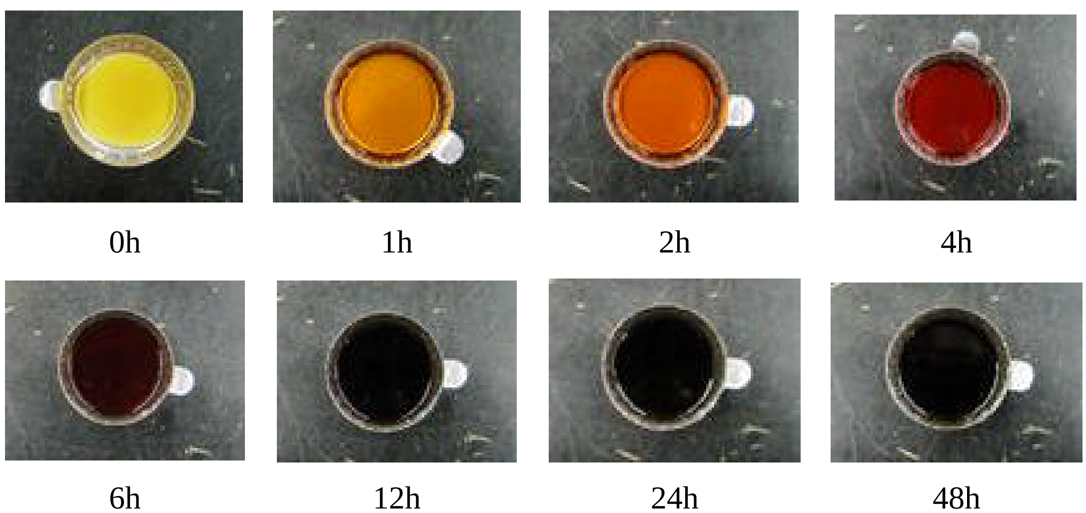


Figure 4-2 Effect of Prolonged Heating on LOF 6500 Antistrip Additive

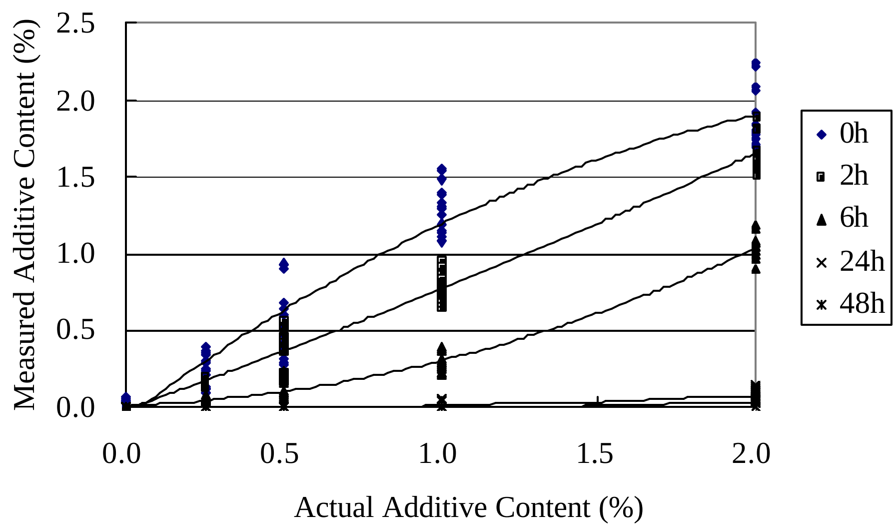


Figure 4-3 Measured LOF 6500 Additive Content in Asphalt Binder as Function of Time using Litmus Test, Measured Additive Content vs. Actual Additive Content

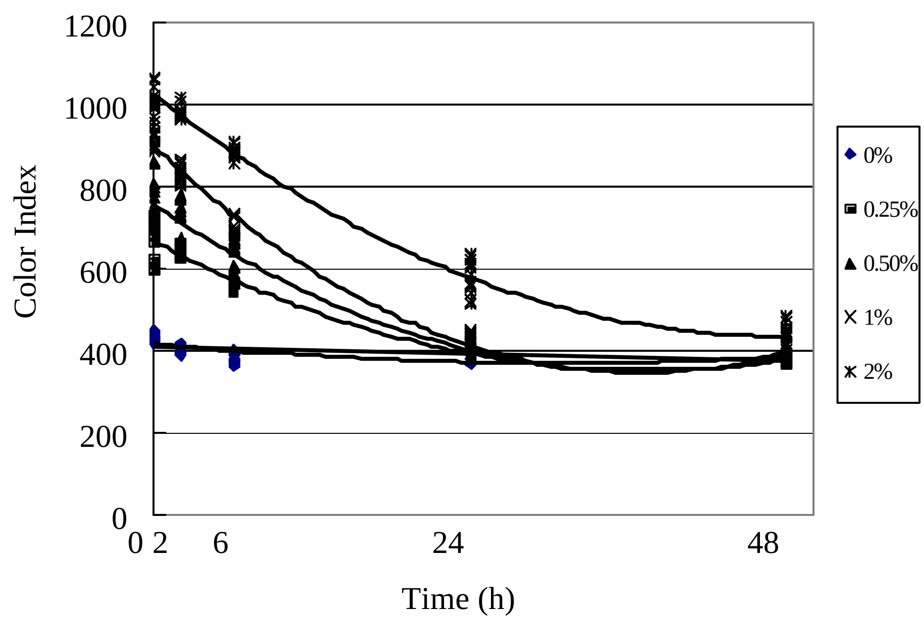


Figure 4-4 Decline of LOF 6500 Additive Content in Asphalt Binder During Prolonged Heating Process using Litmus Test

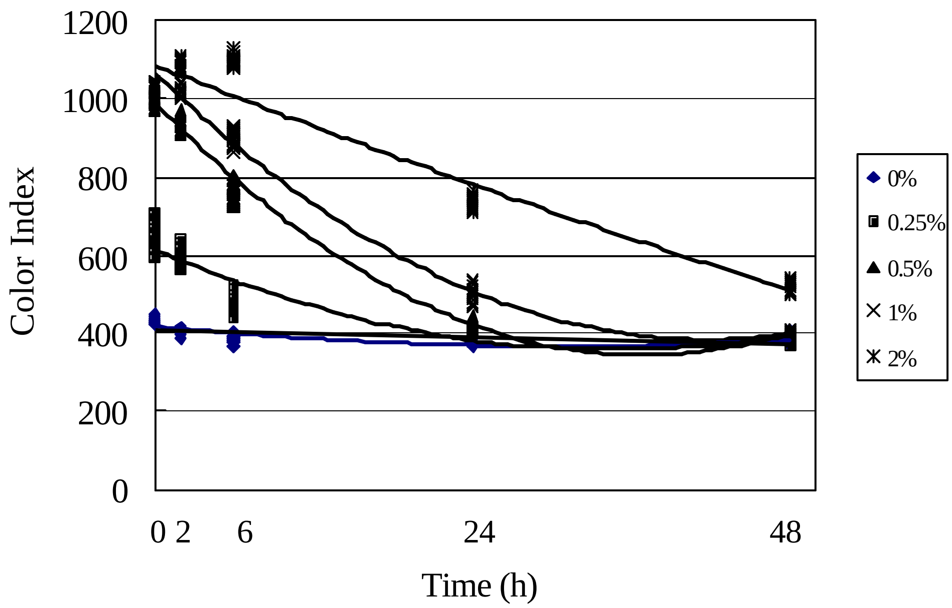


Figure 4-5 Decline of Morlife 2200 Additive Content in Asphalt Binder During Prolonged Heating Process using Litmus Test

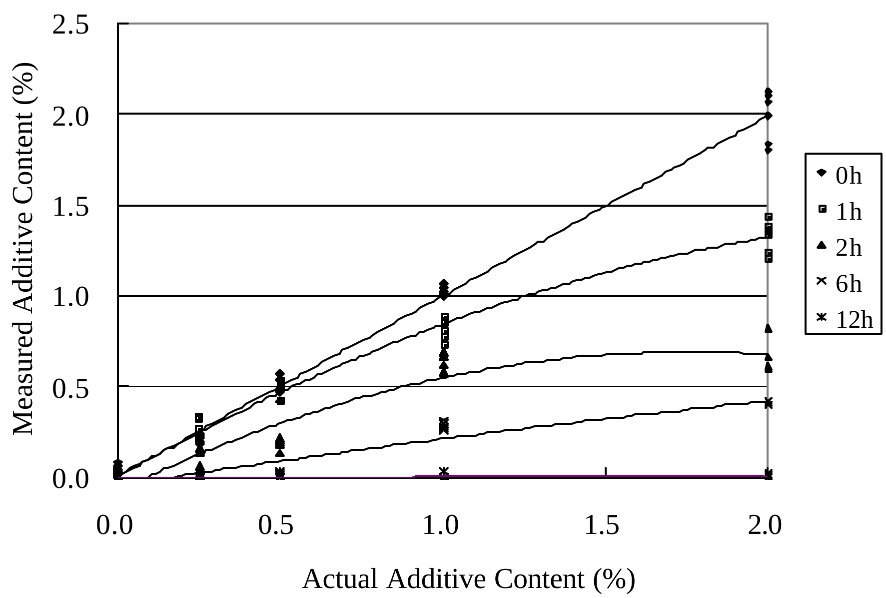


Figure 4-6 Measured LOF 6500 Additive Content in Asphalt Mix I as Function of Time using Litmus Test, Measured Additive Content vs. Actual Additive Content

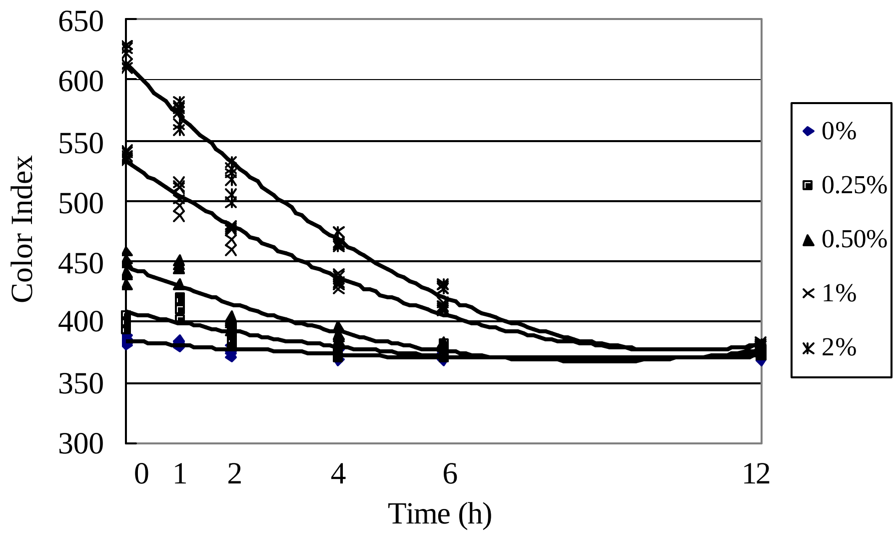


Figure 4-7 Decline of LOF 6500 Additive Content in Asphalt Mix I During Prolonged Heating Process using Litmus Test

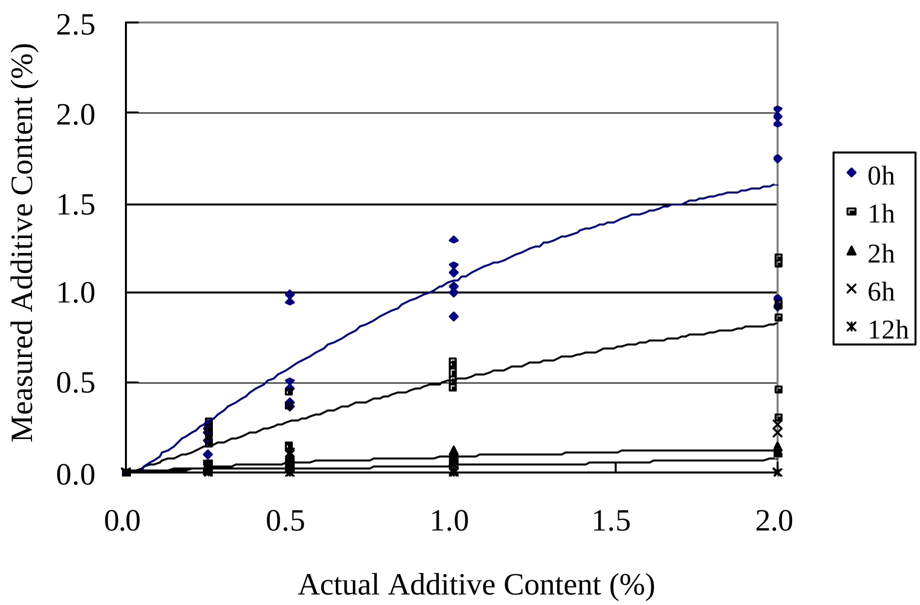


Figure 4-8 Measured Morlife 2200 Additive Content in Asphalt Mix I as Function of Time using Litmus Test, Measured Additive Content vs. Actual Additive Content

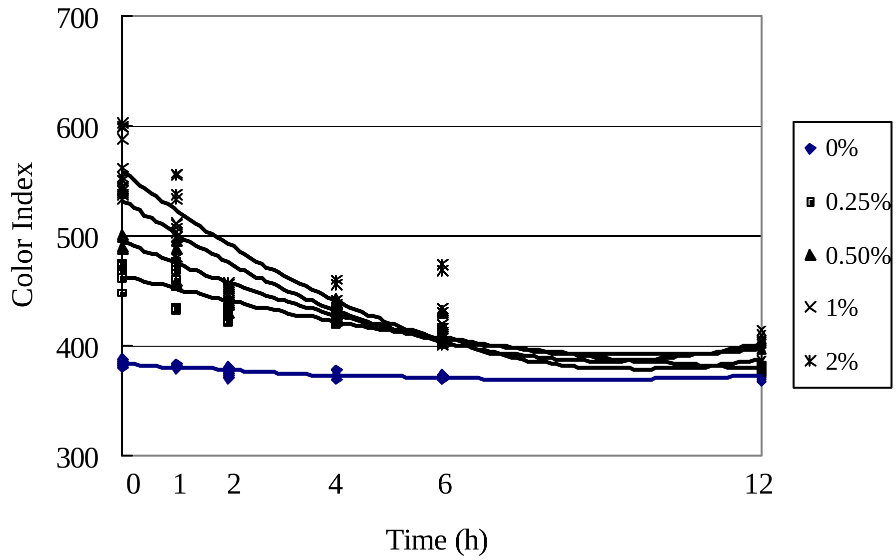


Figure 4-9 Decline of Morlife 2200 Additive Content in Asphalt Mix I During Prolonged Heating Process using Litmus Test

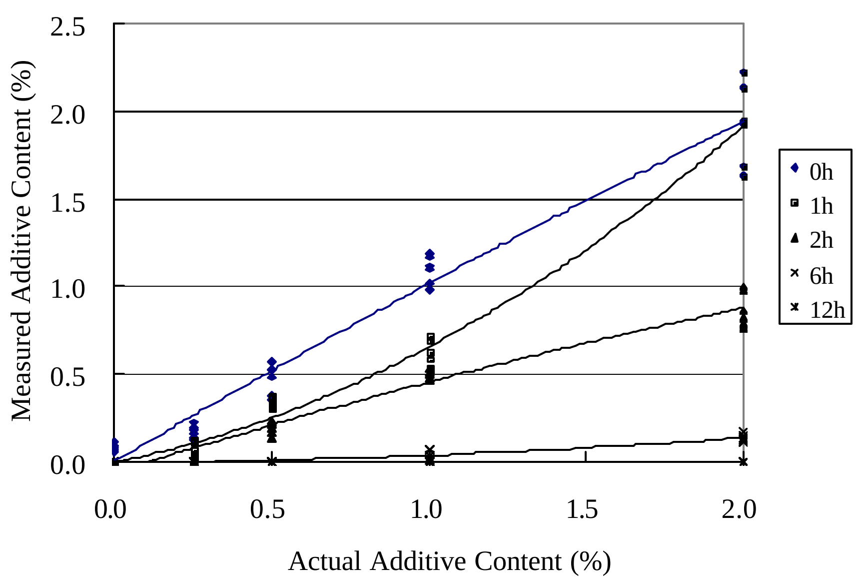


Figure 4-10 Measured LOF 6500 Additive Content in Asphalt Mix II as Function of Time using Litmus Test, Measured Additive Content vs. Actual Additive Content

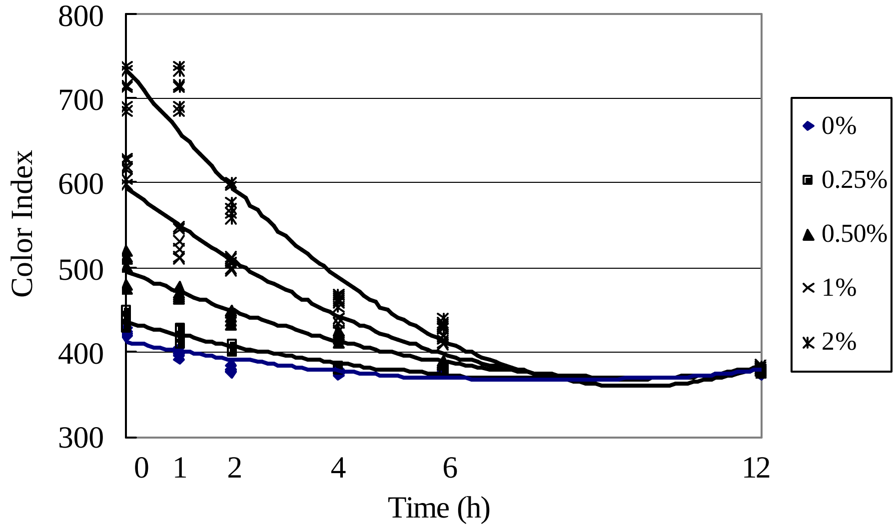


Figure 4-11 Decline of LOF 6500 Additive Content in Asphalt Mix II During Prolonged Heating Process using Litmus Test

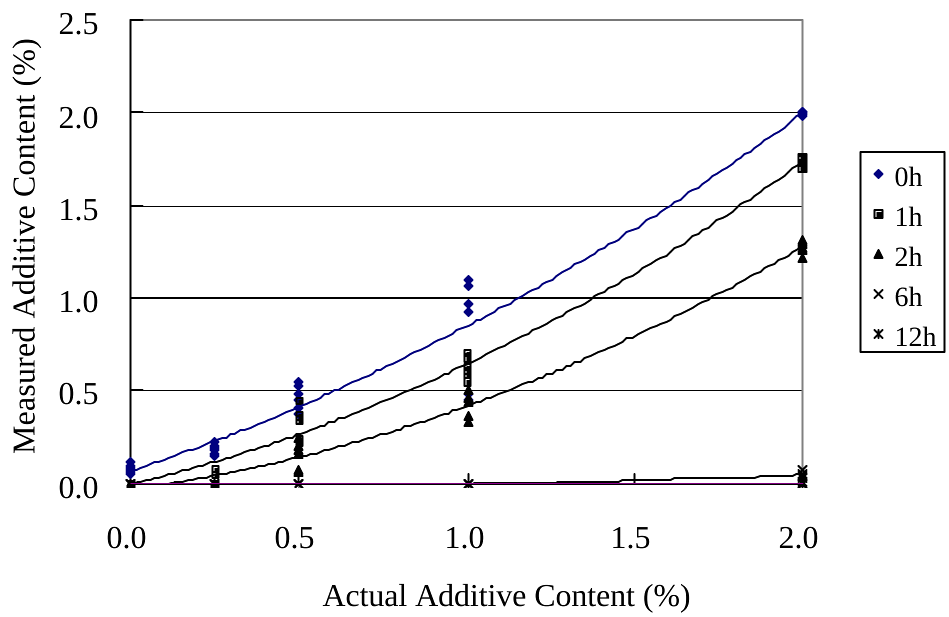


Figure 4-12 Measured Morlife 2200 Additive Content in Asphalt Mix II as Function of Time using Litmus Test, Measured Additive Content vs. Actual Additive Content

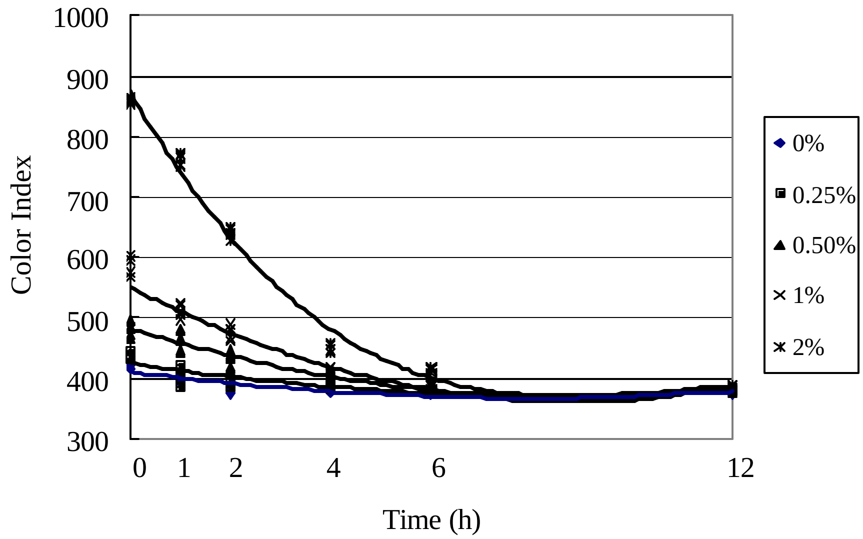


Figure 4-13 Decline of Morlife 2200 Additive Content in Asphalt Mix II During Prolonged Heating Process using Litmus Test

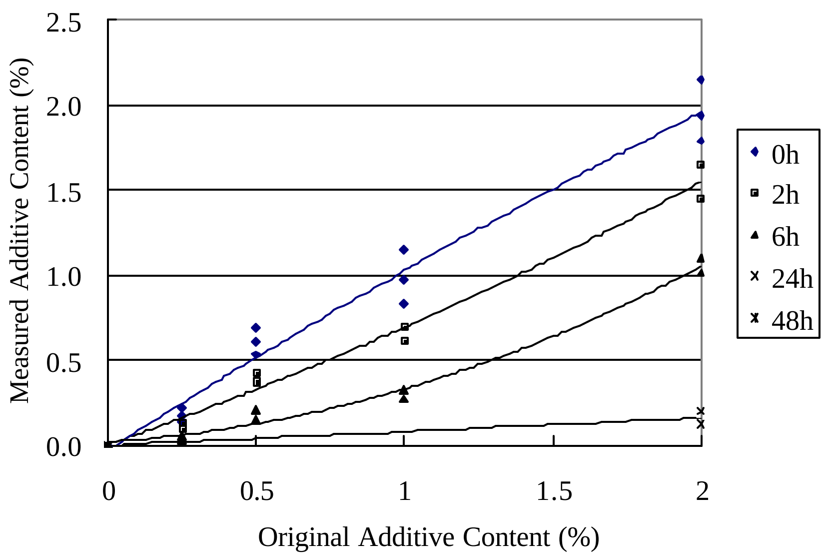


Figure 4-14 Measured LOF 6500 Additive Content in Asphalt Binder as Function of Time using Colorimetric Test, Absorbance vs. Original Additive Content

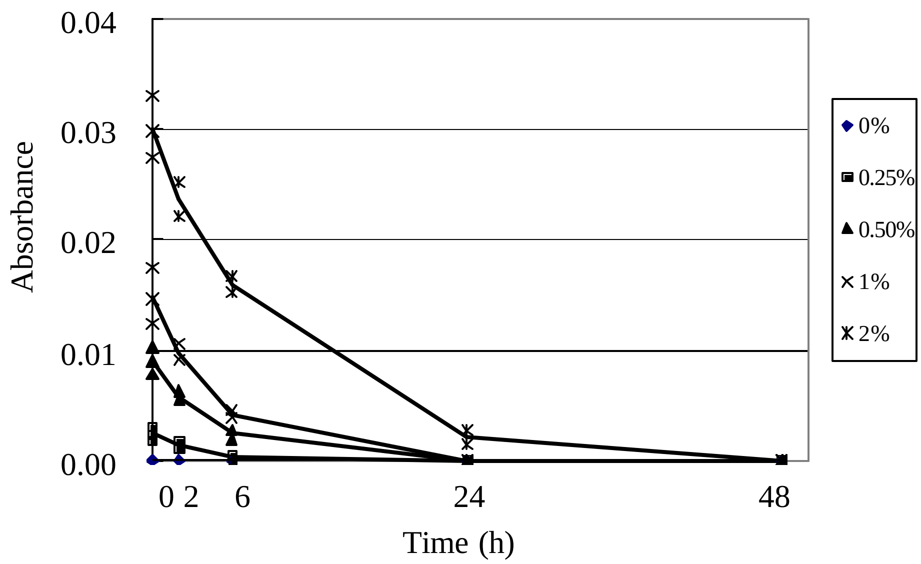


Figure 4-15 Decline of LOF 6500 Additive Content in Asphalt Binder During Prolonged Heating Process using Colorimetric Test

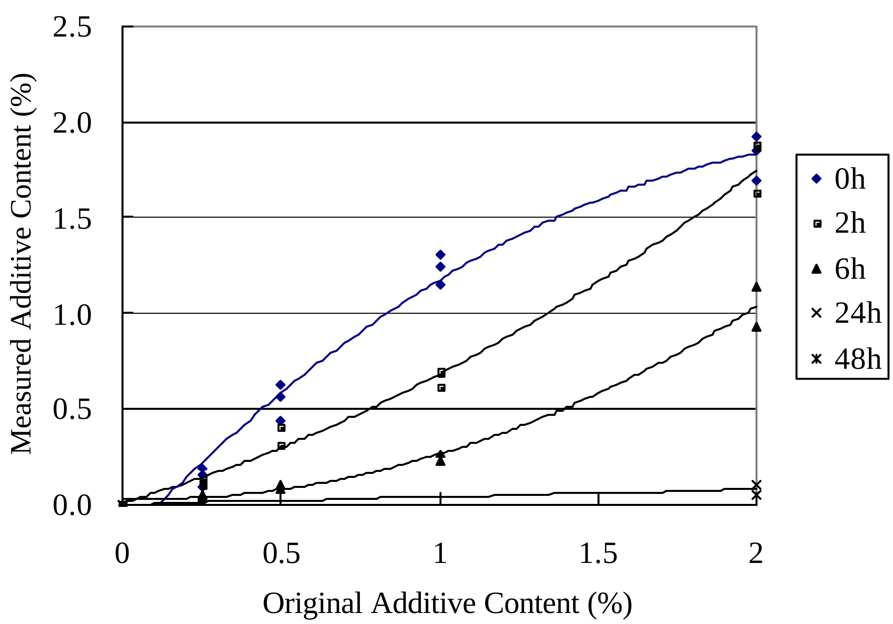


Figure 4-16 Measured Morlife 2200 Additive Content in Asphalt Binder as Function of Time using Colorimetric Test, Measured Additive Content vs. Original Additive Content

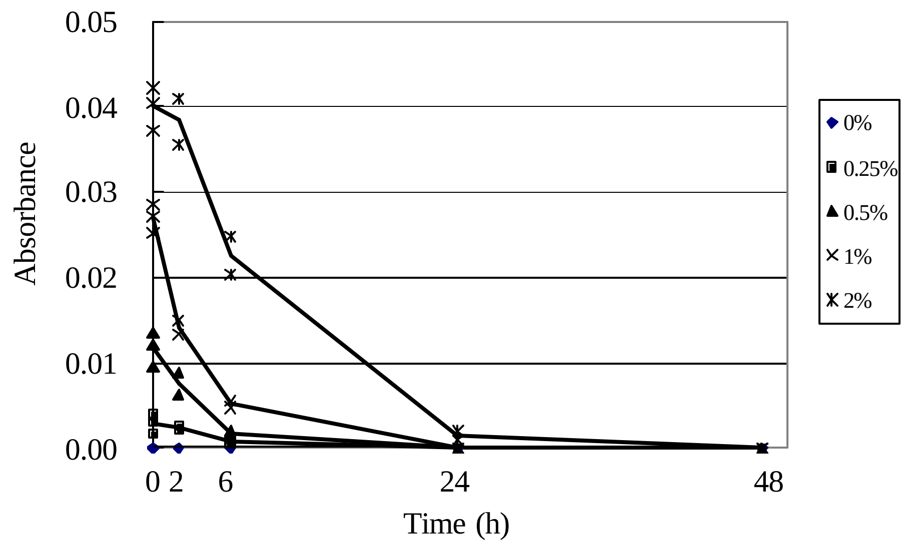


Figure 4-17 Decline of Morlife 2200 Additive Content in Asphalt Binder During Prolonged Heating Process using Colorimetric Test

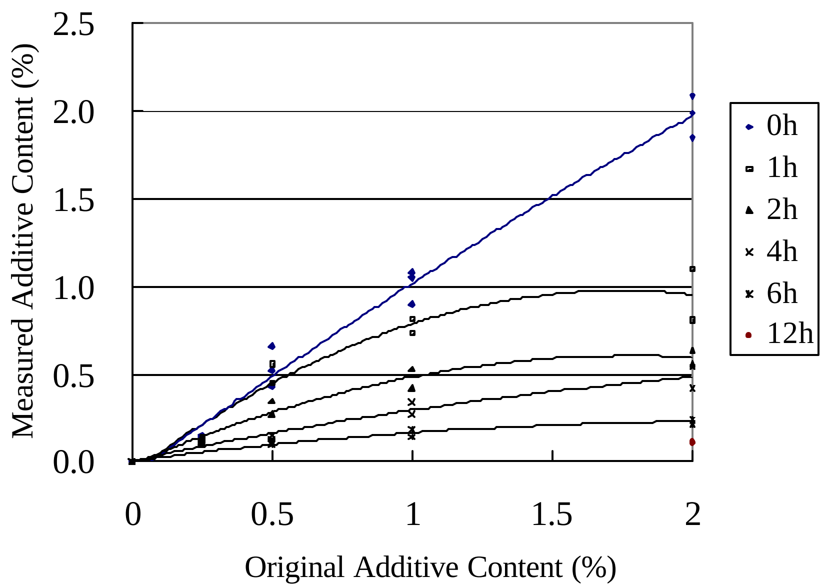


Figure 4-18 Measured LOF 6500 Additive Content in Asphalt Mix I as Function of Time using Colorimetric Test, Measured Additive Content vs. Original Additive Content

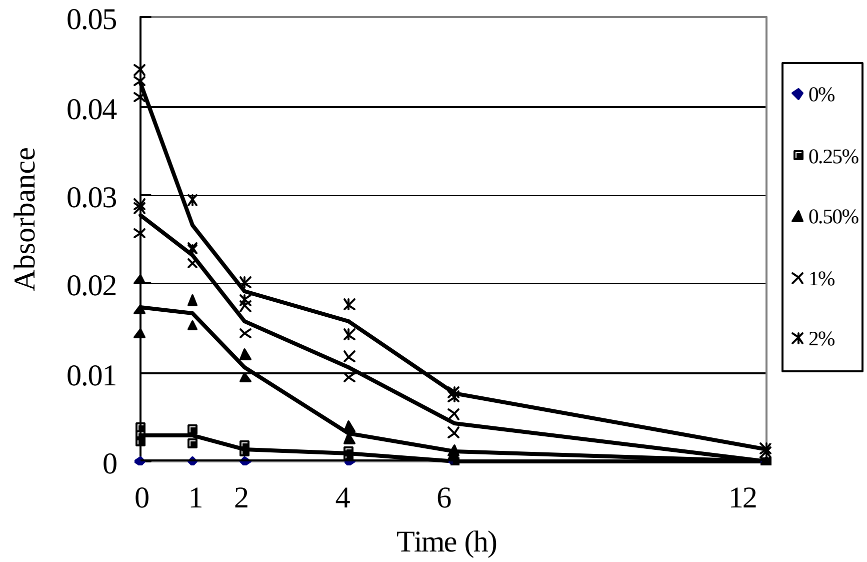


Figure 4-19 Decline of LOF 6500 Additive Content in Asphalt Mix I During Prolonged Heating Process using Colorimetric Test

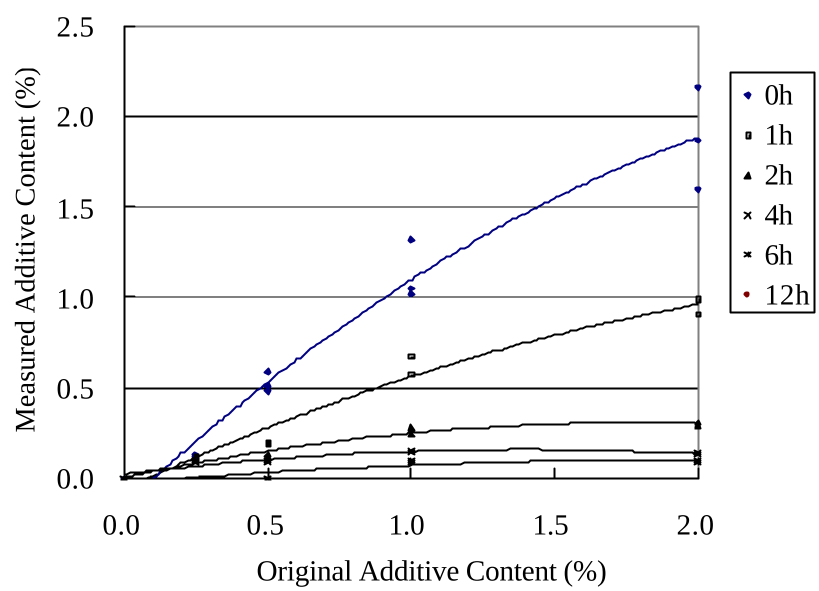


Figure 4-20 Measured Morlife 2200 Additive Content in Asphalt Mix I as Function of Time using Colorimetric Test, Measured Additive Content vs. Original Additive Content

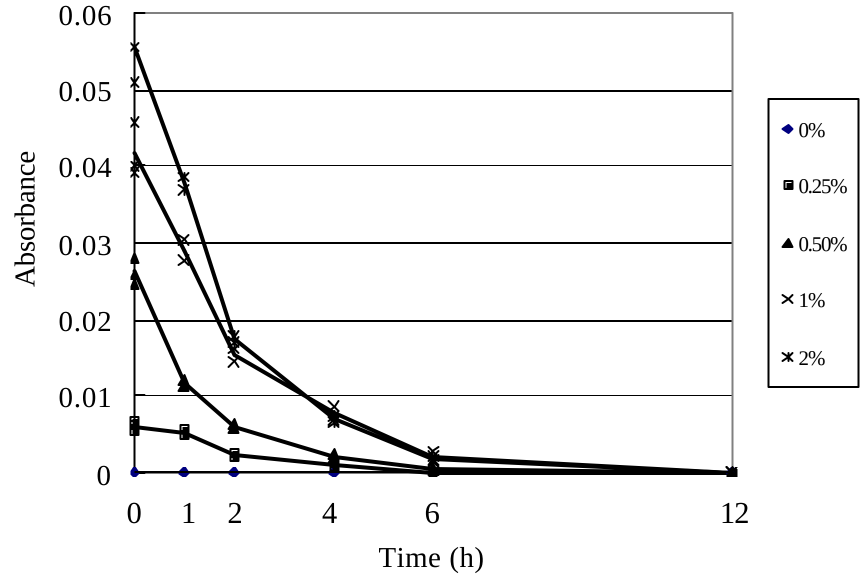


Figure 4-21 Decline of Morlife 2200 Additive Content in Asphalt Mix I During Prolonged Heating Process using Colorimetric Test

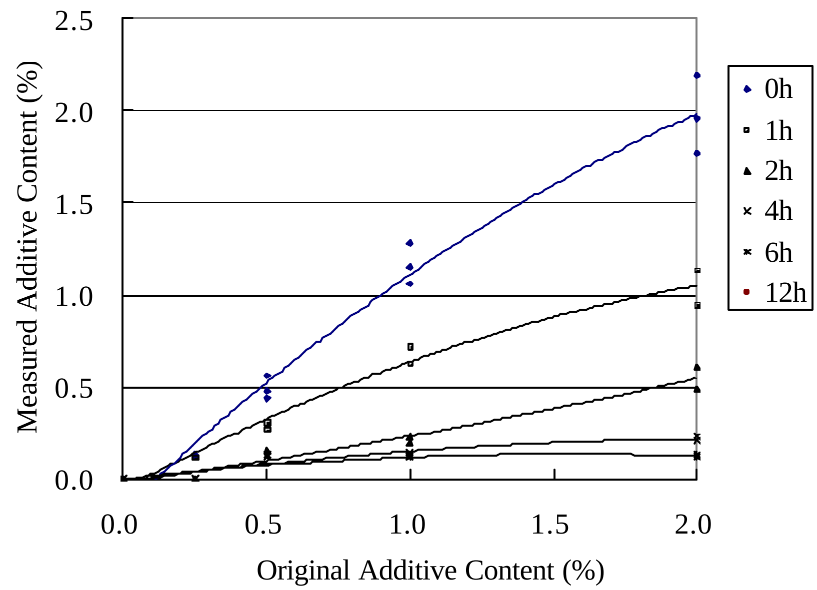


Figure 4-22 Measured LOF 6500 Additive Content in Asphalt Mix II as Function of Time using Colorimetric Test, Measured Additive Content vs. Original Additive Content

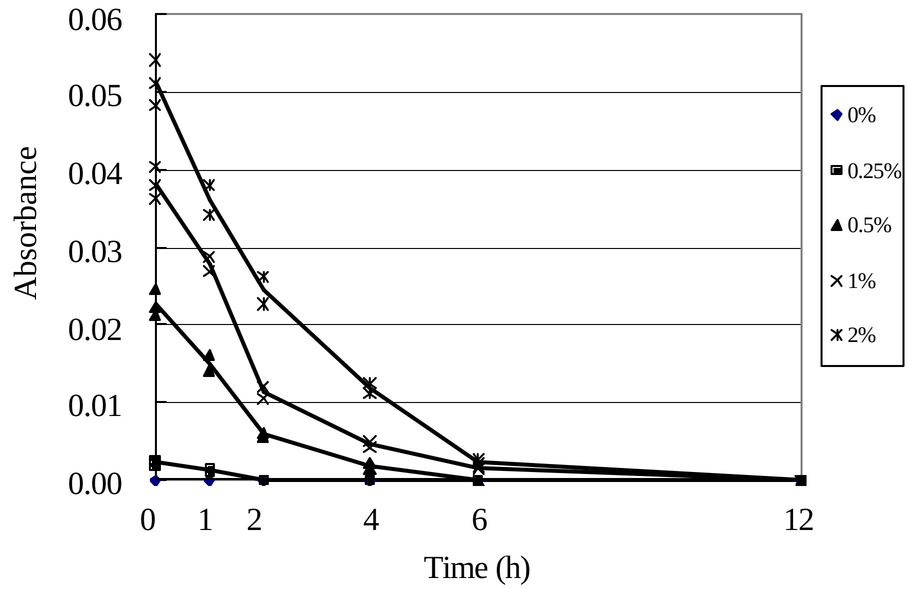


Figure 4-23 Decline of LOF 6500 Additive Content in Asphalt Mix II During Prolonged Heating Process using Colorimetric Test

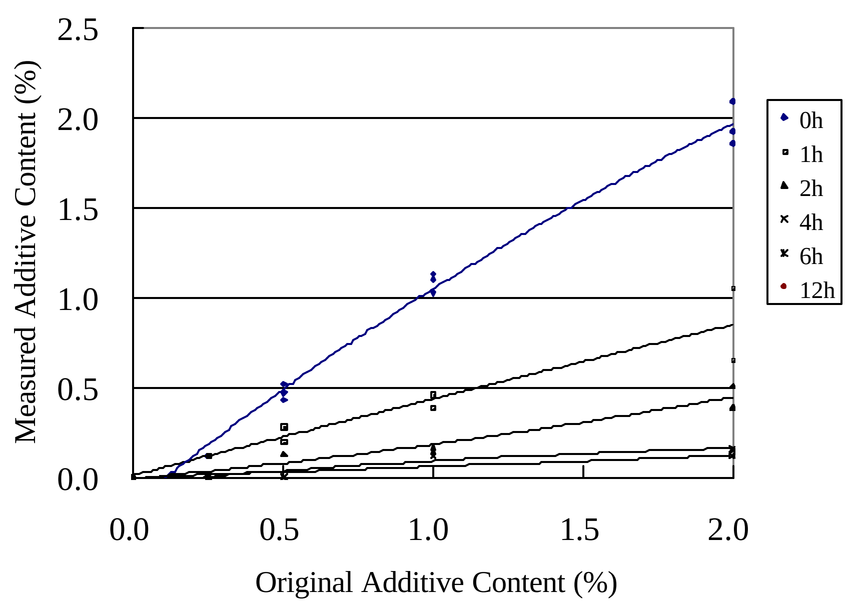


Figure 4-24 Measured Morlife 2200 Additive Content in Asphalt Mix II as Function of Time using Colorimetric Test, Measured Additive Content vs. Original Additive Content

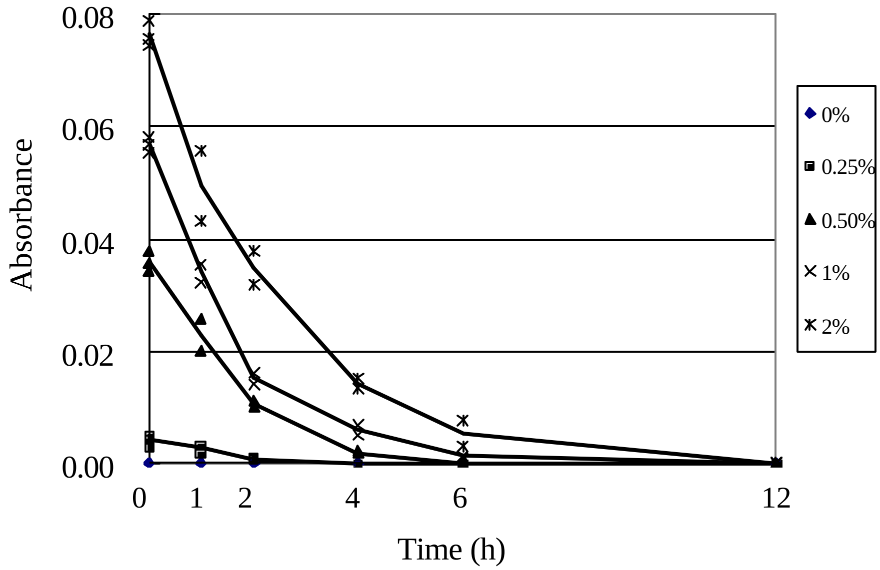


Figure 4-25 Decline of Morlife 2200 Additive Content in Asphalt Mix II During Prolonged Heating Process using Colorimetric Test

5. Summary, Conclusions and Recommendations

5.1 Summary and Conclusions

In this research study, litmus and colorimetric tests were used to quantify amine-based antistrip additives in asphalt binders and mixes. The effect of prolonged heating on antistrip additive content was evaluated for both asphalt binders and mixes. Based on the results of this study, the following observations are offered:

- Both test methodologies rely on the fact that amine-based antistrip additives will volatilize upon heating. If an antistrip additive is very stable at elevated temperatures and does not volatilize, both test methods are likely going to fail not only in determining the antistrip additive content but also in detecting the presence of antistrip additives.
- In both test methods, spectrophotometers were used and the antistrip additive content determination was based on changes in color intensity.
- The litmus test is simpler to conduct and results can be obtained relatively quickly compared to the colorimetric test that requires amines to be extracted into aqueous solution prior to analysis.
- For some antistrip additive types, saturation problems in litmus testing may be encountered at higher additive contents. For this reason it appears that the litmus test may be more suitable for detecting lower additive contents. However, it may be possible to reliably detect higher additive contents by varying the temperature and/or litmus paper exposure time to vapors.
- The colorimetric test seems to be more suitable for detecting higher additive contents and is less sensitive at lower additive contents.

Specific conclusions are as follows:

- Both the litmus and colorimetric tests are capable of detecting and quantifying amine-based antistrip additives in asphalt binders and mixes.
- The change in color intensity was highly correlated to the antistrip additive contents for both test methods.
- Both test methods provided reasonable results when the level of antistrip additive was determined for field samples. However, it needs to be noted that this conclusion is based on testing a single field Mix I with LOF 6500 antistrip additive content of 0.5% by weight of asphalt binder. Additional verification using different mixes, different antistrip additives, and different dosages is needed.
- Based on limited testing conducted on measuring additive contents for field samples in this study, in general, the coefficient of variation (cv) of 5% for the litmus test was lower than the cv of 18.5% for the colorimetric tests. Testing in this study was conducted by a single operator. Testing by multiple operators and in multiple laboratories may produce higher variability in test results.
- The antistrip additive content was found to decrease substantially for asphalt binders and mixes when subjected to prolonged heating periods. For asphalt binders, the measured antistrip additive content was practically zero after 24 to 48 hours of extended heating; for asphalt mixes, the measured antistrip additive content approached zero after 6 to 12 hours of extended heating.

5.2 Recommendations for Future Research

Based on the results of this study, it is evident that there is a loss of organic liquid antistrip additive content in both asphalt binders and mixes when the asphalt binder or mix is subjected to prolonged heating. However, based on the results of this study, it is neither clear nor possible at this time to conclude what effect if any, does the loss of antistrip additive have on the moisture sensitivity of mixes if the loss occurs after mix production, assuming that the specified dosage of antistrip was used during production.

A definitive answer regarding the integrity of asphalt-aggregate bond strength will ensure that NCDOT mixes are not prone to become moisture-susceptible due to loss of organic antistrip through volatilization and/or breakdown in chemical composition. It is therefore recommended that further research be undertaken to study the effect of prolonged heating on asphalt-aggregate bond strength. In addition, the issue of stability and segregation also warrant further investigation.

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Appendix A Data for LOF 6500 and Morlife 2200 in Asphalt Binders and Mixes Using Litmus Test

Table A-1 Data for LOF 6500 Antistrip Additive in Asphalt Binder using Litmus Test, Color Index

Test 1

Content (%)	2h			6h			24h			48h		
0	408	414	416	391	400	401	389	387	392	378	379	384
0.25	662	658	647	568	570	570	396	401	392	370	379	380
0.5	672	663	661	603	605	601	403	406	406	372	376	375
1	801	812	806	659	668	670	420	411	416	380	379	382
2	979	980	972	868	870	854	587	562	559	431	437	433

Test 2

Content (%)	2h			6h			24h			48h		
0	402	397	407	388	378	390	371	370	370	371	372	373
0.25	630	642	633	542	547	543	390	388	391	370	371	370
0.5	741	747	750	651	647	641	411	409	407	374	376	377
1	821	817	825	689	692	687	429	427	429	388	386	386
2	970	968	962	877	872	881	606	603	609	447	454	444

Test 3

Content (%)	2h			6h			24h			48h		
0	409	409	416	376	389	385	397	392	399	405	400	392
0.25	631	622	627	541	552	550	391	388	392	380	381	380
0.5	645	650	651	570	566	562	399	398	399	388	389	389
1	829	821	817	668	679	682	417	415	415	386	388	388
2	977	975	966	882	878	876	521	512	521	412	411	411

Test 4

Content (%)	2h			6h			24h			48h		
0	389	395	387	364	362	370	371	377	369	379	378	376
0.25	657	644	641	579	586	570	410	406	407	380	378	378
0.5	727	721	729	617	609	611	401	403	407	381	387	385
1	833	837	841	700	703	692	436	439	440	401	399	387
2	1017	1006	1005	902	907	886	554	537	560	411	417	419

Test 5

Content (%)	2h			6h			24h			48h		
0	417	411	408	385	376	390	365	369	371	384	377	382
0.25	642	630	637	563	559	555	394	386	388	377	372	378
0.5	767	769	777	669	672	660	422	429	426	382	379	380
1	866	856	850	731	721	726	441	444	447	410	408	407
2	982	986	979	887	881	889	626	631	620	467	478	480

Table A-2 Data for LOF 6500 Antistrip Additive in Asphalt Binder using Litmus Test, Measured Antistrip Additive (%)

Test 1

Content (%)	2h			6h			24h			48h		
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.21	0.20	0.18	0.06	0.06	0.06	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.23	0.21	0.21	0.10	0.10	0.10	0.00	0.00	0.00	0.00	0.00	0.00
1	0.66	0.71	0.68	0.20	0.22	0.23	0.00	0.00	0.00	0.00	0.00	0.00
2	1.63	1.64	1.59	0.97	0.98	0.90	0.08	0.05	0.05	0.06	0.05	0.06

Test 2

Content (%)	2h			6h			24h			48h		
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.14	0.17	0.15	0.04	0.04	0.04	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.43	0.45	0.46	0.19	0.18	0.17	0.00	0.00	0.00	0.00	0.00	0.00
1	0.75	0.73	0.77	0.28	0.28	0.27	0.00	0.00	0.00	0.00	0.00	0.00
2	1.57	1.56	1.52	1.02	1.00	1.04	0.11	0.10	0.11	0.05	0.04	0.05

Test 3

Content (%)	2h			6h			24h			48h		
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.15	0.13	0.14	0.04	0.05	0.04	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.17	0.18	0.19	0.06	0.06	0.05	0.00	0.00	0.00	0.00	0.00	0.00
1	0.78	0.75	0.73	0.22	0.25	0.26	0.00	0.00	0.00	0.00	0.00	0.00
2	1.62	1.61	1.55	1.05	1.03	1.02	0.03	0.03	0.03	0.00	0.00	0.00

Test 4

Content (%)	2h			6h			24h			48h		
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.20	0.17	0.17	0.07	0.08	0.06	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.39	0.37	0.39	0.12	0.11	0.11	0.00	0.00	0.00	0.00	0.00	0.00
1	0.80	0.82	0.84	0.31	0.31	0.28	0.00	0.00	0.00	0.00	0.00	0.00
2	1.90	1.82	1.81	1.16	1.19	1.07	0.05	0.04	0.05	0.00	0.00	0.00

Test 5

Content (%)	2h			6h			24h			48h		
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.17	0.14	0.16	0.05	0.05	0.05	0.11	0.00	0.00	0.00	0.00	0.00
0.5	0.53	0.53	0.56	0.23	0.23	0.21	0.07	0.00	0.00	0.00	0.00	0.00
1	0.96	0.91	0.88	0.40	0.37	0.38	0.05	0.05	0.05	0.00	0.00	0.00
2	1.65	1.68	1.63	1.07	1.04	1.09	0.14	0.15	0.13	0.03	0.03	0.03

Table A-3 Data for Morlife 2200 Antistrip Additive in Asphalt Binder using Litmus Test, Color Index

Test 1

Content (%)	2h			6h			24h			48h		
0	408	414	416	391	400	401	389	387	392	378	379	384
0.25	620	631	639	501	512	521	414	415	414	397	392	394
0.5	961	959	972	801	787	792	437	440	444	401	403	406
1	1041	1019	1020	898	905	892	512	505	509	396	399	401
2	1108	1098	1097	1085	1082	1079	737	720	725	531	520	527

Test 2

Content (%)	2h			6h			24h			48h		
0	402	397	407	388	378	390	371	370	370	371	372	373
0.25	587	576	592	462	475	458	403	410	406	394	392	391
0.5	931	942	944	782	776	769	428	437	431	391	395	395
1	1000	1009	1003	865	878	872	466	468	475	406	401	405
2	1103	1109	1103	1132	1121	1109	749	728	734	524	531	529

Test 3

Content (%)	2h			6h			24h			48h		
0	409	409	416	376	389	385	397	392	399	405	400	392
0.25	578	562	559	473	487	491	397	401	405	370	377	381
0.5	942	933	927	743	752	739	445	441	439	406	404	401
1	1063	1042	1039	927	916	921	538	521	533	400	404	399
2	1085	1065	1070	1092	1090	1087	714	710	721	503	498	507

Test 4

Content (%)	2h			6h			24h			48h		
0	389	395	387	364	362	370	371	377	369	379	378	376
0.25	601	592	607	488	476	469	411	403	405	400	402	391
0.5	939	941	953	762	772	764	431	429	433	397	392	400
1	1022	1019	1010	881	879	876	487	472	476	398	402	401
2	1083	1072	1096	1097	1092	1100	767	741	756	539	540	542

Test 5

Content (%)	2h			6h			24h			48h		
0	417	411	408	385	376	390	365	369	371	384	377	382
0.25	568	571	576	439	441	452	404	406	404	388	381	379
0.5	912	908	922	722	731	729	429	430	430	394	398	401
1	1041	1037	1026	911	909	924	501	497	506	401	407	406
2	1080	1092	1078	1107	1098	1096	742	746	751	511	506	500

Table A-4 Data for Morlife 2200 Antistrip Additive in Asphalt Binder using Litmus Test, Measured Antistrip Additive (%)

Test 1

Content (%)	2h			6h			24h			48h		
0	0.02	0.03	0.03	0.01	0.02	0.02	0.01	0.01	0.01	0.00	0.00	0.00
0.25	0.22	0.23	0.23	0.11	0.12	0.13	0.03	0.03	0.03	0.01	0.01	0.01
0.5	0.52	0.52	0.53	0.38	0.37	0.37	0.05	0.05	0.06	0.02	0.02	0.02
1												
2												

Test 2

Content (%)	2h			6h			24h			48h		
0	0.02	0.01	0.02	0.01	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.19	0.18	0.19	0.07	0.08	0.07	0.02	0.03	0.02	0.01	0.01	0.01
0.5	0.50	0.51	0.51	0.36	0.36	0.35	0.04	0.05	0.05	0.01	0.01	0.01
1												
2												

Test 3

Content (%)	2h			6h			24h			48h		
0	0.03	0.03	0.03	0.00	0.01	0.00	0.01	0.01	0.02	0.02	0.02	0.01
0.25	0.18	0.16	0.16	0.08	0.10	0.10	0.01	0.02	0.02	0.00	0.00	0.00
0.5	0.51	0.50	0.49	0.33	0.33	0.32	0.06	0.05	0.05	0.02	0.02	0.02
1												
2												

Test 4

Content (%)	2h			6h			24h			48h		
0	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.20	0.19	0.20	0.10	0.09	0.08	0.03	0.02	0.02	0.02	0.02	0.01
0.5	0.50	0.50	0.52	0.34	0.35	0.35	0.05	0.04	0.05	0.01	0.01	0.02
1												
2												

Test 5

Content (%)	2h			6h			24h			48h		
0	0.03	0.03	0.02	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.17	0.17	0.18	0.05	0.05	0.06	0.02	0.02	0.02	0.01	0.00	0.00
0.5	0.48	0.47	0.49	0.31	0.32	0.31	0.04	0.04	0.04	0.01	0.02	0.02
1												
2												

Table A-5 Data for LOF 6500 Antistrip Additive in Asphalt Mix I using Litmus Test, Color Index

Test 1

Content (%)	1h		2h		4h		6h		12h	
0	384	380	374	377	377	372	374	372	372	369
0.25	402	410	397	393	376	374	371	373	373	373
0.5	432	444	393	401	390	384	380	379	372	373
1	501	510	476	477	428	430	411	409	383	380
2	582	574	505	498	462	463	430	431	381	382

Test 2

Content (%)	1h		2h		4h		6h		12h	
0	383	382	371	376	369	372	370	372	375	377
0.25	410	416	386	382	371	375	382	379	376	378
0.5	447	450	399	404	396	397	383	380	376	380
1	515	512	480	479	440	437	413	412	379	377
2	559	563	517	522	474	474	428	428	378	380

Test 3

Content (%)	1h		2h		4h		6h		12h	
0	381	379	380	375	377	372	370	369	368	370
0.25	418	420	384	382	376	373	370	372	377	373
0.5	443	431	400	402	388	390	381	382	375	377
1	488	496	459	467	432	433	415	410	379	379
2	576	577	527	532	464	465	428	430	381	377

Table A-6 Data for LOF 6500 Antistrip Additive in Asphalt Mix I using Litmus Test, Measured Antistrip Additive (%)

Test 1

Content (%)	1h		2h		4h		6h		12h	
0	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.21	0.27	0.16	0.13	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.42	0.50	0.13	0.20	0.10	0.04	0.00	0.00	0.00	0.00
1	0.80	0.85	0.67	0.67	0.40	0.41	0.28	0.26	0.03	0.00
2	1.43	1.34	0.82	0.78	0.60	0.60	0.41	0.42	0.01	0.02

Test 2

Content (%)	1h		2h		4h		6h		12h	
0	0.03	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.27	0.32	0.06	0.02	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.51	0.53	0.18	0.22	0.16	0.16	0.03	0.00	0.00	0.00
1	0.88	0.86	0.69	0.68	0.47	0.46	0.29	0.29	0.00	0.00
2	1.20	1.24	0.89	0.92	0.66	0.66	0.40	0.40	0.00	0.00

Test 3

Content (%)	1h		2h		4h		6h		12h	
0	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.33	0.34	0.04	0.02	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.49	0.42	0.19	0.21	0.08	0.10	0.01	0.02	0.00	0.00
1	0.73	0.77	0.58	0.62	0.42	0.43	0.31	0.27	0.00	0.00
2	1.36	1.38	0.95	0.99	0.61	0.61	0.40	0.41	0.01	0.00

Table A-7 Data for Morlife 2200 Antistrip Additive in Asphalt Mix I using Litmus Test, Color Index

Test 1

Content (%)	1h		2h		4h		6h		12h	
0	384	380	374	377	377	372	374	372	372	369
0.25	468	460	434	430	430	432	431	430	376	380
0.5	456	455	439	437	440	442	450	448	402	406
1	507	499	441	440	433	431	420	416	414	405
2	554	556	452	457	431	429	406	412	387	379

Test 2

Content (%)	1h		2h		4h		6h		12h	
0	383	382	371	376	369	372	370	372	375	377
0.25	473	477	424	422	426	424	427	430	379	382
0.5	496	488	443	439	439	435	430	432	398	406
1	512	510	453	447	441	437	434	431	404	410
2	538	533	455	450	459	456	474	469	398	386

Test 3

Content (%)	1h		2h		4h		6h		12h	
0	381	379	380	375	377	372	370	369	368	370
0.25	435	432	427	430	428	420	417	412	380	377
0.5	458	455	436	429	428	422	410	402	384	376
1	503	499	440	438	430	422	408	411	379	381
2	498	480	451	452	428	426	401	404	376	376

Table A-8 Data for Morlife 2200 Antistrip Additive in Asphalt Mix I using Litmus Test, Measured Antistrip Additive (%)

Test 1

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.22	0.17	0.05	0.04	0.04	0.04	0.04	0.04	0.00	0.00
0.5	0.15	0.14	0.07	0.06	0.07	0.08	0.12	0.11	0.00	0.00
1	0.56	0.48	0.08	0.07	0.05	0.04	0.01	0.01	0.00	0.00
2	1.17	1.20	0.13	0.15	0.04	0.03	0.00	0.00	0.00	0.00

Test 2

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.26	0.29	0.02	0.02	0.03	0.02	0.03	0.04	0.00	0.00
0.5	0.45	0.38	0.08	0.07	0.07	0.05	0.04	0.04	0.00	0.00
1	0.61	0.59	0.13	0.10	0.08	0.06	0.05	0.04	0.00	0.00
2	0.94	0.87	0.14	0.12	0.16	0.15	0.26	0.23	0.00	0.00

Test 3

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.05	0.04	0.03	0.04	0.03	0.01	0.01	0.00	0.00	0.00
0.5	0.16	0.14	0.06	0.03	0.03	0.02	0.00	0.00	0.00	0.00
1	0.52	0.48	0.07	0.06	0.04	0.02	0.00	0.00	0.00	0.00
2	0.47	0.31	0.12	0.13	0.03	0.03	0.00	0.00	0.00	0.00

Table A-9 Data for LOF 6500 Antistrip Additive in Asphalt Mix II using Litmus Test, Color Index

Test 1

Content (%)	1h		2h		4h		6h		12h	
0	401	397	377	377	376	375	380	376	376	379
0.25	418	420	399	400	380	382	377	379	377	377
0.5	466	463	437	432	411	415	385	389	379	378
1	522	531	498	500	425	424	410	412	380	384
2	679	695	570	577	467	459	428	430	382	379

Test 2

Content (%)	1h		2h		4h		6h		12h	
0	406	398	376	379	379	374	379	382	379	376
0.25	429	427	401	404	381	384	384	377	377	379
0.5	470	465	442	449	421	419	389	390	378	377
1	545	550	507	512	439	435	415	419	380	381
2	665	657	559	564	453	460	439	435	381	380

Test 3

Content (%)	1h		2h		4h		6h		12h	
0	395	392	380	384	381	372	378	379	378	372
0.25	416	412	401	409	385	384	380	384	377	379
0.5	477	479	448	450	422	424	387	385	382	377
1	512	510	511	505	440	434	420	421	380	378
2	701	699	601	597	469	462	432	428	383	381

Table A-10 Data for LOF 6500 Antistrip Additive in Asphalt Mix II using Litmus Test, Measured Antistrip Additive (%)

Test 1

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.06	0.07	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.31	0.30	0.16	0.14	0.02	0.04	0.00	0.00	0.00	0.00
1	0.58	0.62	0.47	0.48	0.10	0.09	0.01	0.02	0.00	0.00
2	1.58	1.73	0.82	0.86	0.32	0.28	0.11	0.13	0.00	0.00

Test 2

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.12	0.11	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.33	0.31	0.19	0.23	0.07	0.06	0.00	0.00	0.00	0.00
1	0.69	0.72	0.51	0.53	0.17	0.15	0.04	0.06	0.00	0.00
2	1.46	1.39	0.76	0.79	0.25	0.28	0.17	0.15	0.00	0.00

Test 3

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.05	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.37	0.38	0.22	0.23	0.08	0.09	0.00	0.00	0.00	0.00
1	0.53	0.52	0.53	0.50	0.18	0.15	0.07	0.07	0.00	0.00
2	1.79	1.77	1.00	0.97	0.33	0.29	0.14	0.11	0.00	0.00

Table A-11 Data for Morlife 2200 Antistrip Additive in Asphalt Mix II using Litmus Test, Color Index

Test 1

Content (%)	1h		2h		4h		6h		12h	
0	401	397	377	377	384	379	381	378	379	379
0.25	422	417	401	398	390	382	388	390	381	380
0.5	447	444	421	419	389	391	382	386	382	384
1	509	512	483	480	402	400	393	389	388	385
2	768	771	641	645	458	449	416	411	391	389

Test 2

Content (%)	1h		2h		4h		6h		12h	
0	406	398	376	379	381	382	377	379	381	377
0.25	389	392	387	388	388	385	379	382	380	384
0.5	482	480	441	447	403	395	384	389	379	381
1	506	499	467	462	411	408	389	391	385	384
2	754	751	638	629	448	444	421	418	391	383

Test 3

Content (%)	1h		2h		4h		6h		12h	
0	395	392	380	384	380	383	375	378	375	377
0.25	405	409	392	395	389	390	381	388	380	381
0.5	467	464	438	435	414	409	385	389	383	382
1	522	527	491	484	417	420	392	384	391	382
2	772	764	651	648	448	456	415	420	385	389

Table A-12 Data for Morlife 2200 Antistrip Additive in Asphalt Mix II using Litmus Test, Measured Antistrip Additive (%)

Test 1

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.08	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.24	0.22	0.08	0.06	0.00	0.00	0.00	0.00	0.00	0.00
1	0.61	0.63	0.46	0.44	0.00	0.00	0.00	0.00	0.00	0.00
2	1.75	1.76	1.27	1.29	0.31	0.25	0.04	0.01	0.00	0.00

Test 2

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.45	0.44	0.20	0.24	0.00	0.00	0.00	0.00	0.00	0.00
1	0.59	0.55	0.36	0.33	0.01	0.00	0.00	0.00	0.00	0.00
2	1.70	1.69	1.26	1.22	0.25	0.22	0.08	0.06	0.00	0.00

Test 3

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.36	0.35	0.18	0.17	0.03	0.00	0.00	0.00	0.00	0.00
1	0.68	0.71	0.51	0.47	0.05	0.07	0.00	0.00	0.00	0.00
2	1.76	1.74	1.32	1.30	0.25	0.30	0.04	0.07	0.00	0.00

Appendix B Data for LOF 6500 and Morlife 2200 in Asphalt Binders and Mixes Using Colorimetric Analysis

Table B-1 Data for LOF 6500 Antistrip Additive in Asphalt Binder using Colorimetric Test, Absorbance

Content (%)	2h		6h		24h		48h	
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.25	0.0017	0.0012	0.0005	0.0000	0.0000	0.0000	0.0000	0.0000
0.5	0.0062	0.0054	0.0028	0.0020	0.0000	0.0000	0.0000	0.0000
1	0.0091	0.0105	0.0038	0.0045	0.0000	0.0000	0.0000	0.0000
2	0.0251	0.0221	0.0166	0.0152	0.0027	0.0015	0.0000	0.0000

Table B-2 Data for LOF 6500 Additive in Asphalt Binder using Colorimetric Test, Measured Antistrip Additive (%)

Content (%)	2h		6h		24h		48h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.14	0.10	0.06	0.03	0.00	0.00	0.00	0.00
0.5	0.43	0.37	0.21	0.15	0.00	0.00	0.00	0.00
1	0.61	0.70	0.27	0.32	0.00	0.00	0.00	0.00
2	1.65	1.45	1.10	1.01	0.20	0.12	0.00	0.00

Table B-3 Data for Morlife 2200 Antistrip Additive in Asphalt Binder using Colorimetric Test, Absorbance

Content (%)	2h		6h		24h		48h	
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.25	0.0022	0.0027	0.0011	0.0007	0.0000	0.0000	0.0000	0.0000
0.5	0.0088	0.0064	0.0021	0.0016	0.0000	0.0000	0.0000	0.0000
1	0.0134	0.0150	0.0049	0.0057	0.0000	0.0000	0.0000	0.0000
2	0.0357	0.0411	0.0204	0.0249	0.0021	0.0011	0.0000	0.0000

Table B-4 Data for Morlife 2200 Additive in Asphalt Binder using Colorimetric Test, Measured Antistrip Additive (%)

Content (%)	2h		6h		24h		48h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.10	0.13	0.05	0.04	0.00	0.00	0.00	0.00
0.5	0.40	0.30	0.10	0.08	0.00	0.00	0.00	0.00
1	0.61	0.69	0.23	0.26	0.00	0.00	0.00	0.00
2	1.63	1.87	0.93	1.14	0.10	0.05	0.00	0.00

Table B-5 Data for LOF 6500 Antistrip Additive in Asphalt Mix I using Colorimetric Test, Absorbance

Content (%)	1h		2h		4h		6h		12h	
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.25	0.0035	0.0022	0.0017	0.0012	0.0010	0.0007	0.0000	0.0000	0.0000	0.0000
0.5	0.0153	0.0182	0.0121	0.0094	0.0039	0.0027	0.0012	0.0008	0.0000	0.0000
1	0.0223	0.0241	0.0175	0.0144	0.0117	0.0094	0.0054	0.0032	0.0000	0.0000
2	0.0294	0.0239	0.0182	0.0201	0.0142	0.0177	0.0074	0.0079	0.0015	0.0010

Table B-6 Data for LOF 6500 Additive in Asphalt Mix I using Colorimetric Test, Measured Antistrip Additive (%)

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.14	0.12	0.12	0.11	0.11	0.10	0.00	0.00	0.00	0.00
0.5	0.45	0.56	0.35	0.27	0.15	0.13	0.11	0.10	0.00	0.00
1	0.74	0.82	0.53	0.42	0.34	0.27	0.18	0.14	0.00	0.00
2	1.10	0.81	0.56	0.64	0.42	0.54	0.22	0.24	0.11	0.11

Table B-7 Data for Morlife 2200 Antistrip Additive in Asphalt Mix I using Colorimetric Test, Absorbance

Content (%)	1h		2h		4h		6h		12h	
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.25	0.0048	0.0056	0.0020	0.0025	0.0009	0.0012	0.0000	0.0000	0.0000	0.0000
0.5	0.0121	0.0114	0.0062	0.0058	0.0026	0.0018	0.0004	0.0006	0.0000	0.0000
1	0.0277	0.0304	0.0144	0.0162	0.0070	0.0087	0.0027	0.0016	0.0000	0.0000
2	0.0386	0.0369	0.0178	0.0169	0.0074	0.0064	0.0022	0.0014	0.0000	0.0000

Table B-8 Data for Morlife 2200 Additive in Asphalt Mix I using Colorimetric Test, Measured Antistrip Additive (%)

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.11	0.12	0.10	0.10	0.09	0.09	0.00	0.00	0.00	0.00
0.5	0.20	0.19	0.13	0.12	0.10	0.09	0.00	0.00	0.00	0.00
1	0.58	0.67	0.24	0.28	0.14	0.15	0.10	0.09	0.00	0.00
2	0.99	0.91	0.31	0.29	0.14	0.13	0.10	0.09	0.00	0.00

Table B-9 Data for LOF 6500 Antistrip Additive in Asphalt Mix II using Colorimetric Test, Absorbance

Content (%)	1h		2h		4h		6h		12h	
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.25	0.0016	0.0011	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.5	0.0161	0.0142	0.0061	0.0057	0.0022	0.0015	0.0000	0.0000	0.0000	0.0000
1	0.0268	0.0289	0.0105	0.0121	0.0041	0.0050	0.0017	0.0015	0.0000	0.0000
2	0.0379	0.0342	0.0262	0.0227	0.0124	0.0111	0.0026	0.0021	0.0000	0.0000

Table B-10 Data for LOF 6500 Additive in Asphalt Mix II using Colorimetric Test, Measured Antistrip Additive (%)

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.12	0.12	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.31	0.27	0.15	0.15	0.13	0.12	0.00	0.00	0.00	0.00
1	0.63	0.72	0.20	0.23	0.13	0.14	0.12	0.12	0.00	0.00
2	1.14	0.95	0.61	0.49	0.23	0.21	0.13	0.12	0.00	0.00

Table B-11 Data for Morlife 2200 Antistrip Additive in Asphalt Mix II using Colorimetric Test, Absorbance

Content (%)	1h		2h		4h		6h		12h	
0	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.25	0.0033	0.0019	0.0009	0.0005	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
0.5	0.0256	0.0198	0.0102	0.0111	0.0021	0.0017	0.0000	0.0000	0.0000	0.0000
1	0.0322	0.0355	0.0141	0.0162	0.0052	0.0068	0.0009	0.0012	0.0000	0.0000
2	0.0557	0.0431	0.0378	0.0319	0.0151	0.0134	0.0078	0.0029	0.0000	0.0000

Table B-12 Data for Morlife 2200 Additive in Asphalt Mix II using Colorimetric Test, Measured Antistrip Additive (%)

Content (%)	1h		2h		4h		6h		12h	
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	0.12	0.12	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	0.28	0.20	0.13	0.13	0.00	0.00	0.00	0.00	0.00	0.00
1	0.39	0.46	0.15	0.17	0.12	0.12	0.00	0.00	0.00	0.00
2	1.05	0.65	0.51	0.39	0.16	0.15	0.12	0.12	0.00	0.00