



**NCAT Report 98-03**

# **TESTS FOR PLASTIC FINES IN AGGREGATES RELATED TO STRIPPING IN ASPHALT PAVING MIXTURES**

**By**

**Prithvi S. Kandal  
Cynthia Y. Lynn  
Frazier Parker**

**March 1998**



277 Technology Parkway • Auburn, AL 36830

**TESTS FOR PLASTIC FINES IN AGGREGATES RELATED TO  
STRIPPING IN ASPHALT PAVING MIXTURES**

By

Prithvi S. Kandhal  
Associate Director  
National Center for Asphalt Technology  
Auburn University, Alabama

Cynthia Y. Lynn  
Graduate Student  
National Center for Asphalt Technology  
Auburn University, Alabama

Frazier Parker  
Director  
Highway Research Center  
Auburn University, Alabama

NCAT Report 98-03

March 1998

## **DISCLAIMER**

The contents of this report reflect the views of the authors who are solely responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views and policies of the National Center for Asphalt Technology of Auburn University. This report does not constitute a standard, specification, or regulation.

## ABSTRACT

This study was undertaken for determining the best aggregate test method that indicates the presence of detrimental plastic fines in the fine aggregate, which may induce stripping in hot mix asphalt (HMA) mixtures.

Ten fine aggregates representing a wide range of mineralogical compositions and plasticity characteristics were used. Their plasticity characteristics were evaluated by three test methods: (a) sand equivalent value, (b) plasticity index, and (c) methylene blue value. Ten HMA mixtures were made using a common limestone coarse aggregate but these different fine aggregates. Superpave volumetric mix design was used to establish the optimum asphalt contents. Two mix validation tests: (a) AASHTO T283 and (b) the Hamburg wheel-tracking device were used to evaluate the stripping potential of the ten HMA mixtures.

Statistical analysis of the aggregate test data and the mix validation test data shows that the methylene blue test is best related to stripping in asphalt paving mixtures.

**KEYWORDS:** Hot mix asphalt, stripping, moisture damage, plastic fines, fine aggregate, sand equivalent, plasticity index, methylene blue

## **TESTS FOR PLASTIC FINES IN AGGREGATES RELATED TO STRIPPING IN ASPHALT PAVING MIXTURES**

Prithvi S. Kandhal, Cynthia Y. Lynn, and Frazier Parker

### **INTRODUCTION**

The presence of plastic fines in the fine aggregate portion of hot mix asphalt (HMA) may induce stripping in the mix when exposed to water or moisture. This study was undertaken for determining the best aggregate test method or methods that indicate the presence of detrimental plastic fines in the fine aggregate, which may induce stripping in HMA mixtures.

### **REVIEW OF LITERATURE**

The presence of dust and clay coatings on the coarse and/or fine aggregate can inhibit coating between the asphalt cement and aggregate and provide channels for penetrating water (1). The asphalt cement coats the dust coating and is not in contact with the aggregate surface resulting in stripping of the HMA mixture. Such stripping in the field due to excessive dust coating on the aggregate has been reported (2). When the aggregate was washed in the quarry the problem was eliminated. Laboratory studies (3) have also shown improved adhesion characteristics of some dust-contaminated coarse aggregates when washed.

It has also been hypothesized that some very fine clayey material may cause stripping by emulsifying the asphalt binder in the presence of water. Excessive plastic fines can also stiffen the asphalt binder and, therefore, the HMA mixture leading to fatigue cracking.

The sand equivalent test was developed by Hveem (4) to determine the amount of clay in the fine aggregate, which was believed to be detrimental to the performance of HMA mixtures. Plasticity index test results and the amount of the material passing the 75 : m or No. 200 sieve (P200 material) have also been used in judging the quality of the fine aggregates so that the performance of the HMA mixtures is not compromised (5). Clough and Martinez (6) conducted an extensive laboratory investigation and showed a very good correlation between the sand equivalent value (which indicates the amount of clayey fines) of the aggregate and the HMA mixture resistance to stripping.

Some baghouse fines (P200 material) have been determined to be detrimental to the HMA mixtures making them susceptible to moisture-induced damage (7). Aschenbrener (8) studied several HMA mixtures in Colorado and concluded that the sand equivalent test value provides a reasonable indication of the HMA mix susceptibility to moisture-induced damage.

### **MATERIALS, TESTS, AND TEST DATA**

#### **Aggregate Types**

Six fine aggregates were chosen to give a wide range of mineralogical compositions and sand equivalent values. In addition, four fine aggregate blends were made to produce a wider range of sand equivalent values. These fine aggregates were obtained by blending a high plasticity clay with fine aggregates PF-4 (Granite) and PF-6 (Limerock). Table 1 identifies the ten fine aggregates used in this study.

**Table 1. Fine Aggregate Types and Location**

Sample Number	Description	Location
PF-1	Pit Run Natural Sand	Alabama
PF-2	High Calcium Limestone	Alabama
PF-3	Dolomite	Alabama
PF-4	Granite	Georgia
PF-5	Blast Furnace Slag	Alabama
PF-6	Limerock	Florida
PF-7	Granite with 4% High Plasticity Clay	Georgia
PF-8	Limerock with 3.4% High Plasticity Clay	Florida
PF-9	Granite with 2.6% High Plasticity Clay	Georgia
PF-10	Limerock with 1% High Plasticity Clay	Florida

### Aggregate Tests and Results

The following test procedures were used to test the fine aggregates:

- AASHTO T176 Plastic Fines in Graded Aggregates and Soils by use of the Sand (ASTM 2419) Equivalent Test
- AASHTO T90 Determining the Plastic Limit and Plasticity Index of Soils (ASTM D 4318)
- Ohio DOT Methylene Blue Value of Clays, Mineral Fillers, and Fines

#### *Sand Equivalent Test*

The sand equivalent test is used to determine the relative proportions of plastic fines or clay-like material in fine aggregates. Fine aggregate passing the 4.75 mm (No. 4) sieve is placed in a graduated, transparent cylinder which is filled with a mixture of water and a flocculating agent. After agitation and 20 minutes of settling, the sand separates from the clay-like fines, and the heights of sand and sand plus clay are measured. The sand equivalent is the ratio of the height of the sand to the height of sand plus clay times 100. Higher sand equivalent will be obtained in case of a cleaner fine aggregate. Minimum specified sand equivalent values for fine aggregate in HMA range from 26 to 60 (5). The minimum requirement of 45 is most common. The requirement is also based on the type of HMA course such as base and surface course. This test has the advantages that it is quick to perform, requires very simple equipment which can be used with minimal training or experience, and has given reasonably good results.

#### *Plasticity Index*

Plasticity Index is being used by several agencies to measure the degree of plasticity of fines. Plasticity Index (PI) is the difference between the liquid limit and the plastic limit of the material passing 425 : m (No. 40) sieve. ASTM D 1073 (Standard specification for Fine Aggregate in Bituminous Paving Mixtures) and D242 (Standard Specification for Mineral Filler for Bituminous Paving Mixtures) limit the PI of this fraction passing the 425 : m (No. 40) sieve (including the mineral filler) to a value of 4 or less. Some states specify a maximum PI for the P200 material. A review of literature indicates no reported correlation between the PI and the field performance of HMA. Precision data have not been established for liquid limit and plastic limit tests which are based on subjective judgement and experience of the tester.

*Methylene Blue Test*

This French test method is recommended by the International Slurry Seal Association (ISSA) to quantify the amount of harmful clays of the smectite (montmorillinite) group, organic matter and iron hydroxides present in fine aggregate. The test method titled “Determination of Methylene Blue Adsorption Value (MBV) of Mineral Aggregate Fillers and Fines,” is contained in Technical Bulletin 145 of ISSA (9). The principle of the test is to add quantities of a standard aqueous solution of the dye (methylene blue) to a sample until adsorption of the dye ceases.

A representative sample of dry fine aggregate is screened through the No. 200 sieve. The portion of the sample passing the No. 200 sieve is tested for methylene blue value (MBV). Ten grams of the sample are dispersed in 30 grams of distilled water in a beaker. One gram of methylene blue (MB) is dissolved in enough distilled water, to produce 200 ml of solution, so that 1 ml of solution contains 5 mg of methylene blue. This MB solution is titrated step wise in 0.5 ml aliquotes from the burette into the continually stirred fine aggregate suspension. After each addition of MB solution and stirring for one minute, a small drop of the aggregate suspension is removed with a glass rod and placed on a filter paper. Successive additions of MB solution are repeated until the end point is reached. Initially, a well-defined circle of MB-stained dust is formed and is surrounded with an outer ring or corona of clear water. The end point is reached when a permanent light blue coloration or “halo” is observed in this ring of clear water. The MB value of a specific fine aggregate fraction is reported as milligrams of methylene blue per gram of specific fine aggregate fraction such as: MBV = 5.3 mg/g, 0/#200. The MBV expresses the quantity of MB required to cover the total surface of the clay fraction of the sample with a monomolecular layer of the MB. Therefore, the MBV is proportional to the product of the clay content times the specific surface of the clay (10). The methylene blue test is simple and practical, and its cost is reasonable. An Ohio DOT version of the test (given in the Appendix) was used in this study.

Table 2 summarizes measured aggregate properties. Each test was run in triplicate and average values are reported. Specific gravity and water absorption of fine aggregate (AASHTO T84) were determined for mix design purposes only.

**Table 2. Fine Aggregate Test Results**

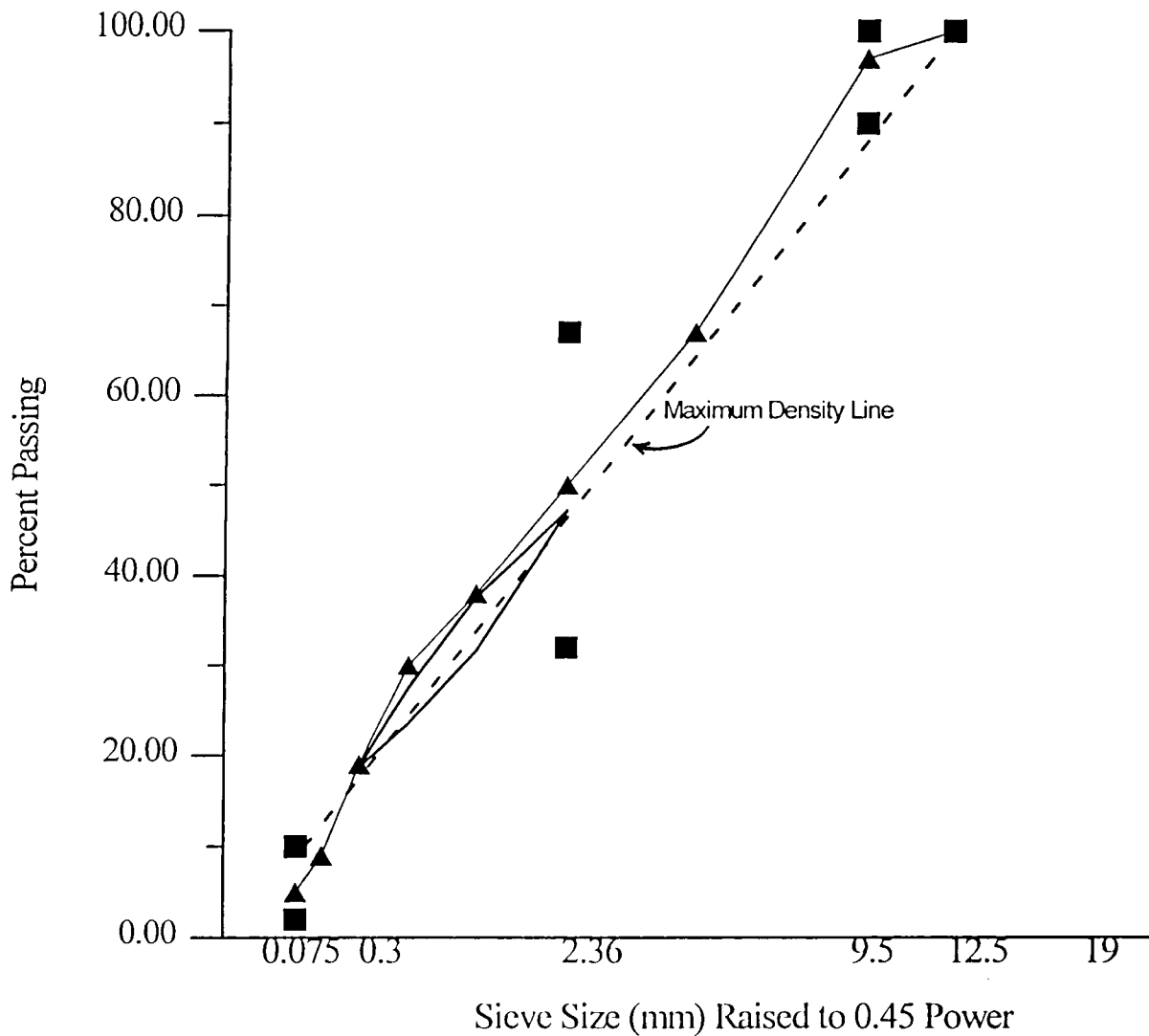
Mix #	PF-1	PF-2	PF-3	PF-4	PF-5	PF-6	PF-7	PF-8	PF-9	PF-10
Test	Natural Sand	Limestone	Dolomite	Granite	Blast Furnace Slag	Limestone	Granite + 4% Clay	Limerock + 3.4% Clay	Granite + 2.6% Clay	Limerock + 1% Clay
Sand Equivalent	24	90	91	58	87	84	39	63	51	74
Plasticity Index (Passing #40)	NP	NP	NP	NP	NP	NP	NP	NP	NP	NP
Plasticity Index (Passing #200)	29	NP	NP	NP	NP	NP	40	40	40	40
Methylene Blue	18.4	1.3	0.3	2.1	2	9.5	80.0	66.0	47.5	26.9
Apparent SG.	2.667	2.744	2.869	2.750	2.711	2.678	2.730	2.672	2.823	2.676
Bulk SG.	2.558	2.665	2.786	2.657	1.623	2.358	2.651	2.363	2.743	2.360
Water Absorption	2.0	1.1	1.1	1.3	1.2	5.1	-	-	-	-

As shown in Table 2, the plasticity index for all of these aggregates, especially the fraction passing 425 : m (No. 40) sieve, was zero (nonplastic), therefore, the two main aggregate tests that were used for comparative purposes were the sand equivalent test and the methylene blue test. As mentioned earlier, the sand equivalence measures the relative amount of clay-sized particles in a fine aggregate. The methylene blue test determines the amount and nature of

potentially detrimental material, such as clay and organic material, that may be present in a aggregate.

*Mixture Tests and Results*

The gradation was held constant for all mixes and is shown in Figure 1. All mixes contained a common limestone coarse aggregate (33%) but different fine aggregates. Superpave volumetric mix design was used to determine the optimum asphalt content to give 4% air voids for mix with each fine aggregate. The mix design data such as optimum asphalt content, voids in the total mix (VTM), and voids in the mineral aggregate (VMA) are given in Table 3.



**Figure 1. Mix gradation**

**Table 3. Superpave Volumetric Mix Design Data**

Mix Designation	Optimum Asphalt Content	VTM	VMA
PF-1	5.5	4.0	14.2
PF-2	5.3	4.0	15.8
PF-3	3.9	4.0	11.9
PF-4	5.2	4.0	14.4
PF-5	6.8	4.0	15.2
PF-6	8.0	4.0	20.6
PF-7	5.2	4.0	14.4
PF-8	8.0	4.0	20.6
PF-9	5.2	4.0	14.4
PF-10	8.0	4.0	20.6

The following two mixture validation tests were used to evaluate the stripping potential of HMA mixtures:

- AASHTO T283 Resistance of compacted Bituminous Mixture to Moisture Induced Damage
- Hamburg Wheel-Tracking Device

Of all the test methods available for evaluating moisture susceptibility, AASHTO T283 (modified Lottman test) is more widely used and its reliability is considered relatively better than numerous other test methods (2). Recent field studies by Aschenbrener (8) indicated that AASHTO T283 could reasonably predict the stripping potential of Colorado aggregates. The moisture susceptibility of the HMA mixtures is quantified in terms of retained tensile strength after water conditioning.

The Hamburg wheel-tracking device (HWTD) was used for additional validation. This device has been used successfully by the City of Hamburg, Germany, the Colorado Department of Transportation (8), and Koch Materials Company's laboratory in Terre Haute, Indiana for identifying aggregates which are susceptible to moisture-induced damage.

The HWTD measures the combined effects of rutting and moisture damage by rolling a steel wheel back and forth across the surface of a HMA slab that is submerged in hot water. The HMA slab is compacted in the laboratory by a mechanically operated compactor called a linear kneading compactor. The slab measures 320 mm in length, 260 mm in width, and 80 mm in thickness. The slab weighs about 15 kg and is compacted to a void content of  $7 \pm 1$  percent. The slab is secured in a reusable steel container with plaster of Paris. The slab is submerged in water which can be heated from  $25^{\circ}\text{C}$  ( $77^{\circ}\text{F}$ ) to  $70^{\circ}\text{C}$  ( $158^{\circ}\text{F}$ ), the standard testing temperature being  $50^{\circ}$  ( $122^{\circ}$ ).

Two slabs can be tested simultaneously with two reciprocating solid steel wheels (diameter 203.5 mm and width 47.0 mm) loaded to 710 N. The length of travel is 230 mm and the average speed is 1.1 km/hour resulting in  $53 \pm 2$  passes/minute.

The testing duration is 20,000 cycles and deformation is recorded and plotted after each cycle. On the cycles versus deformation plot two distinct lines are generally observed. The first line

(rutting line) indicates rutting in the HMA unaffected by stripping. The following second line (stripping line) with a steeper slope indicates rutting due to stripping. The point (number of cycles) where the slope of the rutting line and the slope of the stripping line intersect is called the inflection point. This is the point where stripping is assumed to have been initiated. Inflection point (expressed in terms of number of cycles) is the test parameter of interest for this study. Table 4 contains the average specimen voids in the total mix (VTM) and voids in the mineral aggregate (VMA) information for the slabs used in the Hamburg wheel tracking tests.

**Table 4. VTM and VMA for Hamburg Wheel Tracking Specimens**

Mix Designation	Optimum Asphalt Content	VTM	VMA
PF-1	5.5	6.0	16.0
PF-2	5.3	5.0	15.7
PF-3	3.9	7.1	14.9
PF-4	5.2	4.6	15.0
PF-5	6.8	5.4	15.6
PF-6	8.0	5.0	21.6
PF-7	5.2	5.0	15.5
PF-8	8.0	5.8	22.2
PF-9	5.2	4.2	14.9
PF-10	8.0	5.0	21.5

Table 5 contains the average specimen VTM and VMA information for the specimens used in the AASHTO T283 tests. Mixes PF-6, PF-8, and PF-10 used Florida limerock as the main fine aggregate. Mixes PF-8 and PF-10 only differ from PF-6 in that they contain additional amounts of clay. These three mixes have very high VMA and high optimum asphalt content (Table 3). The only criteria used to determine the optimum asphalt content of these mixes was 4% VTM. It is very likely that this limerock degraded excessively in the gyratory compactor. The resulting degradation will increase the VMA of the mix thus requiring an increased asphalt content in order to achieve the proper VTM. Moreover, the limerock is highly absorptive and, therefore, required additional asphalt binder.

**Table 5. VTM and VMA for AASHTO T283 Specimens**

Mix Designation	Conditioned Specimens		Unconditioned Specimens	
	VTM	VMA	VTM	VMA
PF-1	6.9	16.8	6.9	16.8
PF-2	7.2	17.7	7.4	17.9
PF-3	7.0	14.7	7.0	14.8
PF-4	7.0	17.1	7.0	17.1
PF-5	7.3	17.3	7.2	17.2
PF-6	7.4	23.6	7.3	23.5
PF-7	7.4	17.6	7.3	17.6
PF-8	7.1	23.3	7.1	23.2
PF-9	8.0	18.3	8.4	18.6
PF-10	6.5	23.0	6.5	22.8

Table 6 contains the mixture validation test results.

**Table 6. Mixture Validation Test Results**

Mix Designation	Asphalt Content at 4% Voids	TSR Results, %	Hamburg Wheel Test (Inflection Point)
PF-1	5.5	49.3	5000
PF-2	5.3	85.0	17000
PF-3	3.9	79.1	17500
PF-4	5.2	61.3	6800
PF-5	6.8	47.5	17000
PF-6	8.0	62.0	3800
PF-7	5.2	44.2	5500
PF-8	8.0	48.4	5200
PF-9	5.2	30.8	4300
PF-10	8.0	49.0	2400

## STATISTICAL ANALYSIS OF TEST DATA

Single variable correlation analysis and multiple regression analyses were made using aggregate test values as independent variables and mix validation test values as dependent variables. Correlation matrices were developed using the SAS computer program so that the relationships between aggregate tests and HMA performance parameter (stripping in this case) could be examined in a tabular form. Correlation coefficients, that is, R values were used in the correlation matrices tables because their sign (+ or -) indicates the nature of the relationship between the variables. When linear equations ( $y=a+bx$ ) are shown, coefficients of determination, that is,  $R^2$  values were used since the sign of the “b” coefficient will indicate the nature of the relationship between the independent (x) and the dependent (y) variables.

The forward selection multiple variables procedure given in the SAS program was used to select the aggregate test(s) which is (are) related to HMA stripping. The forward selection procedure begins by finding the variable that produces the optimum one-variable subset, that is, the variable with the largest coefficient of determination or  $R^2$ . In the second step, the procedure finds that variable which, when added to the already chosen variable, results in the largest increase in  $R^2$  and so on. The process continues until no variable considered for addition to the model provides an increase in  $R^2$  considered statistically significant at the specific level ( $P = 0.05$  for this study).

Table 7 contains a correlation between the aggregate tests and mix validation tests. In addition to the comparison to sand equivalent (SE) and methylene blue (MB), the mix validation tests were also compared to variations of the results of these two aggregate tests, in the event that the relationships may not be linear. The variations used in the comparison were the log of each test result, the square of each result, and the inverse of the square of the results.

The TSR is best related to log methylene blue ( $R=-0.79$ ,  $P=0.006$ ). Its correlation with the square of sand equivalent has a  $R=0.67$ ,  $P=0.03$ . Inflection point is also best related to log methylene blue ( $R=-0.82$ ,  $P=0.03$ ). Its correlation with the square of sand equivalent has  $R=0.69$ ,  $P=0.03$ .

**Table 7. Correlation Between Aggregate Properties and Mix Properties<sup>a</sup>**

Aggregate Test	TSR	Inflection Point
Sand Equivalent	0.615 0.06	0.618 0.06
Methylene Blue	-0.639 0.05	-0.552 0.098
(Sand Equivalent) <sup>2</sup>	0.673 0.03	0.686 0.03
(Methylene Blue) <sup>2</sup>	-0.502 0.14	-0.390 0.27
Log(Sand Equivalent)	0.525 0.12	0.528 0.12
Log(Methylene Blue)	-0.794 0.006	-0.825 0.003
1/(Sand Equivalent) <sup>2</sup>	-0.316 0.37	-0.340 0.34
1/(Methylene Blue) <sup>2</sup>	0.538 0.11	0.559 0.09

<sup>a</sup>Top values are correlation coefficients R and bottom values are significance levels P in each cell.

The forward selection procedure in SAS computer program was used to determine if combinations of variables containing methylene blue and sand equivalent values could improve prediction of TSR and inflection point. No combination was found that improved the predictability of the relationships with log MB shown in Table 8. The forward selection procedure did not proceed beyond Step one after selecting the methylene blue test.

Figures 2 and 3 show the plots of log methylene blue versus TSR and inflection point, respectively.

**Table 8. Results of Forward Selection Procedure**

Step	Dependent Variable	Independent Variable	Model	R <sup>2</sup>	P
1	TSR	Log Methylene Blue	TSR = 70.277-6.84(Log Methylene Blue)	0.63	0.006
1	Inflection Point	Log Methylene Blue	Inflection Point=14104.2-2644.4(Log Methylene Blue)	0.68	0.003

Figure 2. Methylene blue vs. TSR

Methylene Blue vs. Inflection Point

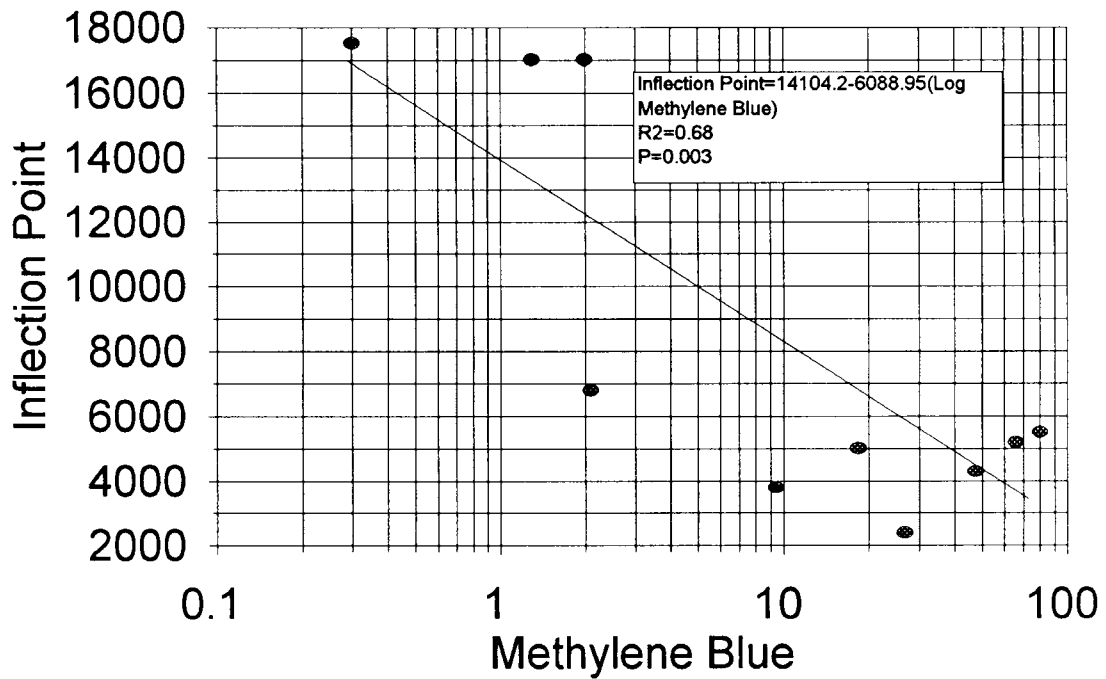


Figure 3. Methylene blue vs. inflection point

## **CONCLUSIONS**

Both TSR and inflection point test data indicate that methylene blue is the fine aggregate test which is best related to stripping of HMA. Therefore, the methylene blue test is recommended to indicate the presence of detrimental plastic fines which may induce stripping in HMA mixtures.

## **REFERENCES**

1. Stuart, KD, "Evaluation of Procedures Used to Predict Moisture Damage in Asphalt Mixtures", FHWA, Report FHWA/RD-86/091, 1986.
2. Kandhal, P.S., "Moisture Susceptibility of HMA Mixes: Identification of Problem and Recommended Solutions," National Asphalt Pavement Association, Quality Improvement Publication 119, December 1992.
3. Balghunaim, F., "Improving the Adhesion Characteristics of Bituminous Mixes by Washing Dust Contaminated Coarse Aggregates," paper presented to the Transportation Research Board, 1990.
4. Hveem, F.N., "Sand Equivalent Test for Control of Materials During Construction," Proceedings, Highway Research Board, Vol. 32, 1953.
5. O'Hara, W.G., "Evaluation of the California Sand-Equivalent Test," Proceedings, Highway Research Board, Vol. 34, 1955.
6. Clough, R.H., and Martinez, J.E., "Research on Bituminous Pavements Using the Sand Equivalent Test," Highway Research Board, Bulletin 300, 1961.
7. Kandhal, P.S., "Evaluation of Baghouse Fines in Bituminous Paving Mixtures," Journal of the Association of Asphalt Paving Technologists, Vol. 50, 1981.
8. Aschenbrener, T., "Comparison of Colorado Component Hot Mix Asphalt Materials with Some European Specifications," Colorado Department of Transportation, Report No. CDOT-DTD-R-92-14, December 1992.
9. International Slurry Seal Association, "Test Method for Determination of Methylene Blue Absorption Value (MBV) of Mineral Aggregate Fillers and Fines," ISSA Bulletin 145, 1989.
10. Hosking, R., "Road Aggregates and Skidding," Transportation Research Laboratory, State-of-the-Art Review 4, HMSO, London, 1992.

## Appendix A

### Standard Method of Test for Methylene Blue Value of Clays, Mineral Fillers, and Fines

#### 1. Scope

- 1.1 This standard provides procedures for determining the amount of potentially harmful fine material (including clay and organic material) present in an aggregate.
- 1.2 The values stated in S1 units are to be regarded as the standard.
- 1.3 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

#### 2. Summary of Test

- 2.1 Methylene Blue solution is titrated into distilled water containing the sample material (passing 75 mm sieve) in increments. A small amount of water containing the sample material and titrated Methylene Blue is removed via a glass rod and dropped onto filter paper. When the sample aggregate can no longer absorb more Methylene Blue, a blue ring is formed on the filter paper.

#### 3. Significance and Use

- 3.1 The Methylene Blue Value determined by this standard can be used to estimate the amount of harmful clays and organic matter present in an aggregate. A large value for Methylene Blue Value indicates a large amount of clay or organic material present in the sample.

#### 4. Apparatus

- 4.1 Amber colored burette of at least 50 ml capacity
- 4.2 Magnetic Mixer with stir bar
- 4.3 Balance sensitive to 0.01g
- 4.4 Glass rod of approximately 250mm length and approximately 8mm diameter
- 4.5 Timer or stop watch
- 4.6 Sieve (75mm) and pan
- 4.7 Volumetric Flask of 1000 ml capacity
- 4.8 Whatman No. 2 filter paper
- 4.9 Glass beakers

- 4.10 Methylene Blue, reagent grade-dated and stored for no more than four months in a brown bottle wrapped with foil in a dark cabinet at lab temperature.
- 4.11 Distilled water at lab temperature

## 5. Sampling

- 5.1 A representative sample of the fine aggregate to be tested is dried to constant weight and screened through the 75mm (No. 200) sieve. The portion passing through the sieve (P200 material) is retained for testing; the rest is discarded.

## 6. Procedure

- 6.1 Weight out 10.0 g (+/- 0.05 g) of the P200 material that has been dried to constant weight and place in beaker.
- 6.2 Add 30g of distilled water and stir with the mixer until the P200 material is uniformly dispersed.
- 6.3 One gram of Methylene Blue is dissolved in enough distilled water to produce 200ml of solution, with each 1 ml of solution contain 5 mg of Methylene Blue.
- 6.4 With the slurry still mixing, fill the burette with the Methylene Blue solution, add 0.5 ml of the solution to the slurry, and stir for one minute.
- 6.5 Remove a drop of the slurry, using the glass stirring rod, and place on the filter paper.
- 6.6 Observe the appearance of the drop on the filter paper. The end point is indicated by the formation of a light blue halo around the drop. Continue adding the Methylene Blue solution to the slurry in 0.5 ml increments with 1 minute stirring after each addition, then testing, until the end point is reached.
- 6.7 After the end point is reached, continue stirring for 5 minutes and retest.  
Note: With experience, the person performing the test can reach the end point more quickly by skipping early increments.

## 7. Calculation

7.1  $MBV = CV/W$

where:

MBV = Methylene Blue Value in mg of solution per g of the P200 material  
C = mg of Methylene Blue/ml of solution  
V = ml of Methylene Blue solution required for titration  
W = grams of dry material

- 7.2 The calculations may be simplified by inserting the mg of Methylene Blue per ml of solution and the grams of dry material.

$$MBV = (5 \times V)/10$$

$$MBV = 0.5V$$

## **8. Precision and Bias**

- 8.1 Precision - No precision has been established for this test.
- 8.2 Bias - No bias can be established because no reference material is available for this test.