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16. Abstract  <p>TxDOT project 0-4524 evaluated the influence of surface energy of aggregates and binders on the resistance of asphalt mixtures to moisture damage. The results from this research lead to the development of a three-tier approach to assess the moisture damage resistance of asphalt mixtures. This approach is based on testing and evaluating the physical and/or mechanical properties of the constituent materials, the fine aggregate mixture, and full asphalt mixture. In the first tier, an energy-based parameter termed the energy ratio (ER) is calculated using the surface energy measurements. This parameter is used as a screening tool to select binders and aggregates that have good resistance to moisture damage. The second and third tiers rely on measuring the mechanical properties of the fine aggregate mixture and full asphalt mixture, respectively.</p> <p>This report documents the results of an implementation project of the testing methods and analysis approaches of the 0-4524 project. This implementation project included (a) providing training on the developed experimental and analysis methods, (b) conducting measurements of the surface energy of binders, additives, and aggregates, and (c) developing a database of surface energy measurements. This database will be useful as a diagnostic tool for finding the cause of poor moisture damage resistance in mixes and to suggest remedies through modification with anti-strip agents, lime, polymers, other additives, or through a change of materials in extreme cases.</p>			
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DEVELOPMENT OF A DATABASE FOR SURFACE ENERGY  
OF AGGREGATES AND ASPHALT BINDERS

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# CHAPTER 1. INTRODUCTION AND BACKGROUND

## 1.1 BACKGROUND

Moisture damage is a major form of pavement distress resulting in high maintenance costs of state and federal highways. Moisture damage can be defined as loss of strength and durability due to the presence of moisture at the binder-aggregate interface (adhesive failure) or within the binder (cohesive failure). Current laboratory procedures for assessing moisture sensitivity of an hot mix asphalt (HMA) rely on comparing mechanical properties of unconditioned specimens with moisture conditioned specimens. Although this approach is helpful in comparative analysis of the moisture susceptibility of various mixes, it does not focus on measuring the fundamental material properties related to the mechanisms described above. As such, the results cannot be used to explain causes for poor or good performance and do not provide feedback into the process of redesigning better performing mixes. It is therefore necessary to supplement the mechanical properties normally measured with fundamental properties that affect physical adhesion between the asphalt and aggregate and the propensity to lose this bond in the presence of water.

As part of the TxDOT 0-4524 project, the researchers developed a three-tier approach to assess the moisture damage resistance of asphalt mixtures. The three tiers are based on testing and evaluating the physical and/or mechanical properties of the constituent materials, the sand-asphalt mixture, and full asphalt mixture. In the first tier, an energy-based parameter termed the energy ratio (ER) is calculated using the surface energy measurements. This parameter is used as a screening tool to select binders and aggregates that have good resistance to moisture damage. In the second tier, the dynamic mechanical analysis (DMA) of fine aggregate matrix specimens consisting of asphalt binder and fine portion of aggregates is used to evaluate moisture susceptibility. Finally, in the third tier the moisture susceptibility of the full mixture is evaluated. The testing at the second and third tiers yields a crack growth index that is a function of fundamental material properties. The DMA is useful to evaluate moisture susceptibility of the materials without being influenced by mixture design and internal structure distribution.

The evaluation of the full mixture is necessary, however, in order to verify that the mixture design and internal structure distribution are optimized to improve the resistance to moisture damage. At the end of the 0-4524 project, researchers recommended a tentative range of values for the parameters determined at each of the three steps.

## **1.2 IMPLEMENTATION STUDY**

It was decided to implement the findings from the 0-4524 project, starting with the first tier. This implementation project was developed in order to transfer the know-how on test and analytical methods that are used to characterize the fundamental properties of aggregate and binder that influence the resistance to moisture damage. The objectives of this implementation project were to:

- Provide training on the developed experimental and analysis methods.
- Measure surface energy of binders, additives, and aggregates. These measurements will be used to more completely analyze surface energies of typical materials; to evaluate the effects of additives and modifiers on the surface energies of commonly used asphalts; and to evaluate the changes in aggregate and asphalt surface energies due to changes in refinery processes and geological strata over time.
- Incorporate surface energy measurements from the previous work and from this implementation project into a database. This database will be useful as a diagnostic tool to determine the cause of poor moisture damage resistance in mixes and to suggest remedies through modification with anti-strip agents, lime, polymers, other additives, or through a change of materials in extreme cases.

The aforementioned objectives were achieved by accomplishing the following tasks in this project.

**Task 1:** Training on the use of Wilhelmy plate device, Universal Sorption Device, and Dynamic Mechanical Analyzer testing protocols and analysis methods.

**Task 2:** Measurements of the surface energy of aggregates and binders that are typically used in the State of Texas.

**Task 3:** Analysis of changes in surface energies of aggregate samples from the same source. This task is aimed at determining the variability in aggregate surface energy over time and due to changes in the geological strata within a given source.

**Task 4:** Analysis of changes in surface energies of binder samples from the same supplier. This task is aimed at determining the variability in binder surface energy due to changes in the binder source and modification process.

**Task 5:** Development of an updated database of aggregate and binder surface energies. This database will be valuable for the selection of aggregate and binder combinations with very good resistance to moisture damage. Also, it will be used later to verify the analysis methods developed in project 0-4524 through comparison between the predicted resistance to moisture damage and field performance.

**Task 6:** Purchase equipment to measure surface energy on aggregates and asphalt binders for installation at CST Central laboratory, and provide training and technical assistance on the operation of the equipment.

Chapter 2 presents a summary on the aforementioned tasks in seriatim.



## **CHAPTER 2. SUMMARY OF TASKS**

This chapter presents a brief summary on each of the tasks accomplished in this implementation project.

### **2.1 TRAINING ON THE USE OF SURFACE ENERGY AND DMA EQUIPMENT**

Measurement of surface free energy of materials and mechanical properties of the fine aggregate matrix (FAM) are the first two steps in the three-tier approach to mixture characterization and design. This task was designed to provide hands on training to TxDOT personnel who may want to run these tests at the CST laboratory. The first training was provided to six TxDOT personnel on March 8, 2007, at the Texas Transportation Institute laboratory in College Station. The training included the following three tests:

1. Dynamic mechanical analysis (DMA) to evaluate the mechanical properties of the FAM.
2. Test method to determine the surface free energy of asphalt binders using the Wilhelmy plate device.
3. Test method to determine the surface free energy of aggregates using the Universal Sorption Device (USD).

The one-day training included a theory session that included a background on each of the test methods, a detailed explanation of the test procedure, and demonstration of the analytical techniques to interpret results from each of these test methods. Appendix A of this report includes the training material. The theory session was followed by hands on training with each one of the test methods.

## **2.2 MEASUREMENT OF SURFACE ENERGY OF AGGREGATES AND BINDERS**

### **2.2.1 Surface Free Energy of Aggregates**

The Universal Sorption Device (USD) was used to determine the surface free energy of aggregates. The surface free energy values for a total of 16 different types of aggregates were compiled in this research. Of these 16 different types of aggregates, the surface free energy components of five different types of aggregates were determined by obtaining samples from the same source at two different points in time. This resulted in a total testing of 21 aggregates. The objective of this exercise was to evaluate changes in the surface free energy characteristics amongst different batches of aggregates obtained from the same source. Section 2.3 presents more details on this objective that was accomplished in Task 3.

At least three different replicates for each aggregate type were tested with each one of the three probe liquids. All tests were conducted in accordance with the draft protocol that was developed following the NCHRP 9-37 project and TxDOT 0-4524 project. The draft protocols are included in Appendix B of this report. This appendix also includes guidelines to use the spreadsheet to compute the surface free energy components using the spreading pressures determined using the USD test method. Table 1 enumerates the list of aggregates that were included in Tasks 2 and 3. The results from these tests are included in the database of surface energy values. Section 2.5 presents further details regarding this database.



**Table 1. List of Aggregates Used for Surface Energy Measurement.**

<b>Number</b>	<b>Mineralogy</b>	<b>Quarry</b>	<b>Procurement Date</b>
1a	Granite	Snyder, OK	May-04
1b	Granite	Snyder, OK	Jun-07
2	Quartzite	Jones Mill, AK	May-04
3a	Sandstone	Sawyer, OK	May-04
3b	Sandstone	Sawyer, OK	Jun-07
4	Silicious River Gravel	Prescott, AK	May-04
5	Limestone	Bridgeport, TX	May-04
6	Limestone	Spore "Bucyrus" OH	May-04
7	River Gravel	Melco, OH	May-04
8a	Crushed River Gravel	Murphy, TX	Oct-03
8b	Crushed River Gravel	Murphy, TX	Jun-07
9a	Crushed Limestone	Brownwood, TX	May-04
9b	Crushed Limestone	Brownwood, TX	Jun-07
10a	Crushed Traprock	Knippa, TX	Apr-04
10b	Crushed Traprock	Knippa, TX	Jun-07
11	Sandstone	Sawyer, OK	Feb-06
12	Limestone	New Braunfels, TX	Jul-06
13	Sandstone	Brownlee, TX	May-06
14	Limestone	Hunter, TX	May-06
15	Limestone	Odessa, TX	Unknown
16	Granite	Herndon, VA	Jun-02
17	Limestone	Material Reference Library (MRL)	Oct-07
18	Granite	Material Reference Library (MRL)	Oct-07

Note: Aggregate with alpha-numerical numbering such as 1a and 1b indicate that the aggregates were sampled from the same source at two different times for surface energy measurement.

### **2.2.2 Surface Free Energy of Asphalt Binders**

The Wilhelmy plate device was used to determine the surface free energy of asphalt binders. The surface free energy values for a total of 44 different types of neat and modified asphalt binders were compiled in this project. Of these, 24 binders were obtained from seven different manufacturers and included base as well as polymer

modified binders. The remaining 20 binders were obtained by modifying a subset of the 24 binders used before. The modifications included aging or addition of liquid anti-strip agents. Of these 24 binders, the surface free energy components of 21 different types of asphalt binders was determined by obtaining samples from the same source at two different points in time. The objective of this exercise was to evaluate the changes in surface free energy characteristics among different batches of binder obtained from the same producer (discussed in Section 2.4).


At least three different replicates for each binder type were tested with each one of the five probe liquids. All tests were conducted in accordance with the draft protocol that was developed following the NCHRP 9-37 project and TxDOT 0-4524 project. The draft protocols are included in Appendix C of this report. Table 2 lists asphalt binders that were included in Tasks 2 and 4. The results from these tests are included in the database of surface energy values. Section 2.5 presents further details regarding this database.

**Table 2. List of Asphalt Binders Used for Surface Energy Measurement.**

Source	Source 1 (2004)			Source 2 (2004)				Source 3 (2004)			Source 4 (2004)					Source 5 (2004)			Source 6 (2004)			Source 7 (2004)		
PG Grade and Modifier	64-22	70-22	76-22	64-22	70-22	76-22	76-22	64-22	70-22	76-22	64-22	70-22	76-22	76-28	76-28	64-22	70-22	76-22	64-22	70-22	76-22	64-22	70-22	76-22
	B	S	S	B	S	TRS	S	B	S	TRS	B	S	S	S	S	B	S	S	B	S	S	B	S	S
Unaged Binder																								
SAFT Aging																								
PAV Aging																								
Anti-strip 1																								
Anti-strip 2																								

Source	Source 1 (2007)			Source 2 (2007)				Source 4 (2007)					Source 5 (2007)			Source 6 (2007)			Source 7 (2007)					
PG Grade and Modifier	64-22	70-22	76-22	64-22	70-22	76-22	76-22	64-22	70-22	76-22	70-28	76-28	64-22	70-22	76-22	64-22	70-22	76-22	64-22	70-22	76-22	64-22	70-22	76-22
	B	S	S	B	S	TRS	S	B	S	S	S	S	B	S	S	B	S	S	B	S	S	B-S	S	S
Unaged Binder																								
SAFT Aging																								
PAV Aging																								
Anti-strip 1																								
Anti-strip 2																								

Note: Descriptions provided by binder suppliers: B = Base asphalt binder; S = SBS modifier; TRS = Tire rubber & SBS modifiers.

 Indicates surface energy measurements were made on that specific binder

### 2.3 VARIATION IN SURFACE ENERGY OF AGGREGATES FROM THE SAME SOURCE

A database of any material property is valid and meaningful for future use only as long as the subject material property does not change over time. The objective of Task 3 was to determine the change in surface free energy of an aggregate obtained from the same source but mined and produced at different points in time. In other words, the objective of this task was to determine whether the surface free energy of aggregates changed significantly due to differences in the geologic location over time during the quarrying operations. Five different aggregates were selected for this task (Table 1). These aggregates represented different lithology including granite, limestone, sandstone, river gravel, and traprock. Two samples of each of these aggregates were obtained from the same location with a minimum interval of two years between each of the two samples. The surface energy components of the aggregates were determined using the procedures described before.

Variation in the surface characteristics of aggregates over time was determined based on the spreading pressure of three different probe liquids. The comparison was not made directly based on the surface energy components for the following two reasons. First, the error propagation model to determine standard deviations in the computed surface free energy components based on the standard deviations in the measured spreading pressures is not well established. Second, the exact same numbers of replicates were not used with different probe vapors tested with the same aggregate. A z-test was conducted to determine whether or not the average spreading pressure changed between two aggregate samples collected at different points in time. The null hypothesis in this case is  $H_0 : \mu_1 = \mu_2$ . Table 3 lists the z-statistic for different aggregates and whether the spreading pressure was the same over time (accept null hypothesis) or whether it was different (reject null hypothesis) at significance of  $\alpha$  of 0.05.

**Table 3. Z-Statistic for Change in Spreading Pressure of Various Probe Liquids  
with Aggregate Samples Obtained at Different Times.**

Aggregate	Years in which the Two Samples Were Obtained	Probe Liquid					
		Hexane		MPK		Water	
		Z	Conclusion	Z	Conclusion	Z	Conclusion
Limestone	2004 – 2007	-1.67	SAME	-7.65	DIFFERENT	-1.47	SAME
Traprock	2004 – 2007	-0.36	SAME	-20.03	DIFFERENT	-2.51	DIFFERENT
Sandstone	2004 – 2007	-5.78	DIFFERENT	-5.35	DIFFERENT	-5.60	DIFFERENT
Gravel	2004 – 2007	-3.12	DIFFERENT	-2.44	DIFFERENT	-2.40	DIFFERENT
Granite	2004 – 2007	-15.60	DIFFERENT	-7.24	DIFFERENT	-0.94	SAME

In most cases, the spreading pressure of the probe liquids with the same aggregate had changed due to the difference in time. Since the time gap between the testing of these aggregates was two years or more, it is possible that the changes in spreading pressure may be due to other reasons. For example, it is possible to have a bias in the values due to different operators over time (or even the same operator) over a prolonged duration of time. However, if bias in measurements is the only cause of this change, then it is reasonable to expect that at least the rank order of different aggregates based on their spreading pressures will not change over time.

In order to examine this, the Kendall-Tau rank correlation coefficient was computed for each spreading pressure. This coefficient compares the rank order of the five aggregates with respect to any given spreading pressure. The value of this coefficient ranges from -1 to +1, where -1 indicates that the rankings are perfectly inverse of each other, and +1 indicates that the rankings are perfectly correlated with each other. A coefficient value of 0 means that there is absolutely no correlation between the rankings. In this case, a coefficient value of about 0.8 or higher would indicate that the rank order of spreading pressure for different aggregates does not change significantly with time. Table 4 presents the Kendall Tau correlation coefficient between aggregate samples at different points in time based on the spreading pressures with each of the three liquids.

**Table 4. Kendall Tau Correlation Coefficient  
for Aggregate Spreading Pressures.**

<b>Probe Liquid</b>	<b>Kendall – Tau Rank Correlation Coefficient</b>
Hexane	0.6
MPK	0.8
Water	0.8

From Tables 3 and 4, it is evident that the surface properties of aggregates obtained from the same source are changing over time. However, the ranking of these aggregates based on their polar components (reflected by spreading pressures) did not change significantly. It is important to reiterate here that the polar components of the aggregate are the most significant contributors to determining the moisture damage potential for any different pair of aggregate and asphalt binder. Therefore, the surface energy components of the aggregates available from a database may be used for future materials selection with some caution. It is also recommended that the surface energy of the aggregates be measured and monitored based on samples collected more frequently than two to three years.

#### **2.4 VARIATION IN SURFACE ENERGY OF BINDERS FROM THE SAME SOURCE**

The objective of Task 4 was to determine the change in surface free energy of an asphalt binder obtained from the same manufacturer, with same PG grade, and similar modification if any, but produced at a different point in time or batch. Twenty-one different asphalt binders were selected for this task (Table 2). Two samples of each binder were obtained in 2004 and 2007, respectively. After obtaining the samples the binders were stored in air tight containers at low temperatures to ensure that any permanent change to the asphalt binder due to oxidation was minimal. The surface energy components of the binders were determined using the procedures described before.

Unlike aggregates, there is a well-defined error propagation model to determine the standard deviation in each component based on the standard deviation in the measured contact angles with different probe liquids. Variations in the surface free energy of the binders were determined based on the average value and standard deviation of each surface energy components. A z-test was conducted to determine whether or not the average surface energy components changed between two otherwise similar asphalt binder samples collected at different points in time. The null hypothesis in this case is  $H_0 : \mu_1 = \mu_2$ . Table 5 lists the z-statistic for different asphalt binders and whether the surface energy components were similar over time (accept null hypothesis) or different (reject null hypothesis) at significance of  $\alpha$  of 0.05.

In all cases, at least one of the three surface free energy components changed due to changes in the batch used for the test sample. As in the case of aggregates, it is possible that the change in surface energy of the binder may be due to a bias in the measurements. As before a rank correlation coefficient was used to estimate whether the rank order of different binders based on their surface energy components were preserved over time despite the changes in the absolute values. Table 6 presents the Kendall Tau correlation coefficient between asphalt binders sampled at two different points in time based in their surface energy components.

**Table 5. Z-Statistic for Change in Surface Energy Components of Asphalt Binders Obtained at Two Different Points in Time.**

Asphalt Binder	Surface Energy Component					
	Non-Polar (LW)		Acid (+)		Base (-)	
	Z	Conclusion	Z	Conclusion	Z	Conclusion
Source 1: 64-22 B	-20.7	DIFFERENT	-6.3	DIFFERENT	-1.5	SAME
Source 1: 70-22 S	-10.3	DIFFERENT	-4.0	DIFFERENT	-5.5	DIFFERENT
Source 1: 76-22 S	-19.8	DIFFERENT	-4.1	DIFFERENT	-4.2	DIFFERENT
Source 5: 64-22 B	-13.0	DIFFERENT	-6.8	DIFFERENT	-8.7	DIFFERENT
Source 5: 70-22 S	-1.9	SAME	-3.5	DIFFERENT	-15.5	DIFFERENT
Source 5: 76-22 S	-9.3	DIFFERENT	-6.1	DIFFERENT	-8.1	DIFFERENT
Source 7: 64-22 B	-0.5	SAME	-0.2	SAME	-3.8	DIFFERENT
Source 7: 70-22 S	-9.8	DIFFERENT	-10.1	DIFFERENT	-13.6	DIFFERENT
Source 7: 76-22 S	-26.9	DIFFERENT	-11.0	DIFFERENT	-7.7	DIFFERENT
Source 6: 64-22 B	-0.2	SAME	-1.5	SAME	-4.3	DIFFERENT
Source 6: 70-22 S	-8.1	DIFFERENT	-6.0	DIFFERENT	-2.2	DIFFERENT
Source 6: 76-22 S	-21.9	DIFFERENT	-7.5	DIFFERENT	-5.8	DIFFERENT
Source 2: 64-22 B	-6.4	DIFFERENT	-1.6	SAME	-7.7	DIFFERENT
Source 2: 70-22 S	-11.5	DIFFERENT	-6.6	DIFFERENT	-9.4	DIFFERENT
Source 2: 76-22 S	-0.8	SAME	-6.8	DIFFERENT	-6.1	DIFFERENT
Source 2: 76-22 TRS	-11.9	DIFFERENT	-2.9	DIFFERENT	-8.6	DIFFERENT
Source 4: 64-22 B	-6.9	DIFFERENT	-0.3	SAME	-6.4	DIFFERENT
Source 4: 70-22 S	-7.1	DIFFERENT	-4.3	DIFFERENT	-2.4	DIFFERENT
Source 4: 76-22 S	-10.2	DIFFERENT	-4.9	DIFFERENT	-9.1	DIFFERENT
Source 4: 70-28 S	-9.1	DIFFERENT	-4.0	DIFFERENT	-1.3	SAME
Source 4: 76-28 S	-1.3	SAME	-4.4	DIFFERENT	-8.0	DIFFERENT

**Table 6. Kendall Tau Correlation Coefficient for Asphalt Surface Energy Components.**

Surface Energy Component	Kendall – Tau Rank Correlation Coefficient
Non-Polar	0.09
Acid	0.09
Base	0.44

From Tables 5 and 6 it is evident that the surface energy components of similar asphalt binders from the same source were significantly different from one batch to another. Unlike the polar components of the aggregates, the rank correlation coefficient for asphalt binders based on their polar components was very poor. This indicates that the surface free energy and concomitant ranking of asphalt binders are both susceptible to significant changes when samples are obtained from different batches. It is recommended that the surface energy of the asphalt binder be measured much more frequently than the aggregates.

## **2.5 DATABASE OF SURFACE ENERGY OF AGGREGATES AND BINDERS**

A database of surface energy values for all the materials tested in Tables 1 and 2 was prepared. The database was in the form of a Microsoft Excel<sup>®</sup> spreadsheet and includes several user-friendly features such as determination of the energy parameters for materials selection. Appendix D provides more details on using this spread sheet to add / delete information and interpret results from existing data. The electronic database with the complete data from this project will accompany this report as product P1.

## **2.6 SURFACE ENERGY EQUIPMENT INSTALLATION AND TRAINING**

A Wilhelmy plate device was purchased and installed at the TxDOT CST laboratory in Cedar Park campus in August 2008. A second round of training was provided to the project coordinator and laboratory personnel at the TxDOT facility. The training included specimen preparation and testing using the Wilhelmy plate device. The device to measure surface free energy of aggregates was not purchased per the instructions from TxDOT.



## CHAPTER 3. SUMMARY AND CONCLUSIONS

A total of 21 aggregates and 44 different types of asphalt binders were included in this implementation project. The three surface energy components of these materials were determined and included in a user-friendly electronic database. The electronic database allows the user to add/modify/delete surface free energy properties of aggregates and asphalt binders. The database also computes the adhesion characteristics for different combinations of asphalt binders and aggregates that can be selected by the user.

The surface energy components of two samples of five different aggregates were determined in this project. The two samples were obtained from the same aggregate source but at least two years apart. Results indicate that although the surface free energy components of the aggregates from the same source may change over time, the rank order of the aggregates based on their polar components did not change significantly.

The surface energy components of two samples for each of the 21 different asphalt binders were determined in this project. The two samples were obtained from the same manufacturer but from different batches of production. Results indicate that the surface free energy components of the asphalt binder are likely to change significantly with changes in the batch of production. Also, the rank order of the asphalt binders based on their surface energy components changed significantly over time.

It is recommended that the surface energy components of asphalt binders and aggregates be measured and monitored over time. Surface energy of asphalt binders must be measured and incorporated into the database more frequently than the aggregate.



**APPENDIX A:  
TRAINING MATERIAL**

This appendix presents the training material that was used during the demonstration and training on the use of DMA and surface energy equipment.



# Dynamic Mechanical Analyzer

Training as a part of the Implementation Program

## OUTLINE

- Mix Design  
(FAM – Fine Aggregate Matrix)
- Sample Preparation
- Test Method
- Data Analysis

## MIX DESIGN

Sample	FAM Design				FAM Design				FAM Design				FAM Design			
	Filler	Binder	FA	MA	Filler	Binder	FA	MA	Filler	Binder	FA	MA	Filler	Binder	FA	MA
1	100	0	0	0	100	0	0	0	100	0	0	0	100	0	0	0
2	100	0	0	0	100	0	0	0	100	0	0	0	100	0	0	0
3	100	0	0	0	100	0	0	0	100	0	0	0	100	0	0	0
4	100	0	0	0	100	0	0	0	100	0	0	0	100	0	0	0
5	100	0	0	0	100	0	0	0	100	0	0	0	100	0	0	0
6	100	0	0	0	100	0	0	0	100	0	0	0	100	0	0	0
7	100	0	0	0	100	0	0	0	100	0	0	0	100	0	0	0
8	100	0	0	0	100	0	0	0	100	0	0	0	100	0	0	0

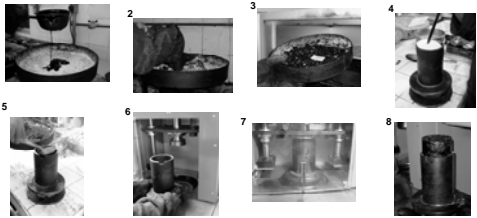
F/A - filler and binder proportion by volume  
 A/A - binder plus filler and fine aggregates proportion in mass  
 Example

## MIX DESIGN

- Alternative and simpler FAM design procedure being developed.
- More representative of the mastic in the mixture
- Requires fewer assumptions
- Lab trials in progress to determine any adjustments if required to make specimen compactible in SGC

## SAMPLE PREPARATION

### CONVENTIONAL 6" SGC SAMPLE PREPARATION

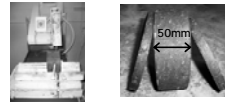


- At least 3min of mixing is usually necessary
- 2h short term aging at compaction temperature
- 150mm
- Wait around 10min before remove the sample from the mold

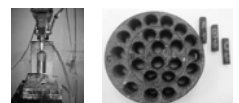
## SAMPLE PREPARATION




### SAWING PROCESS




### CORING PROCESS



## SAMPLE PREPARATION




**Gmb DETERMINATION**



WEIGHT IN WATER

**GLUING PROCESS**




## TEST METHOD

### BOHLIN RHEOMETER

- CREEP / CREEP RECOVERY
- VISCOMETRY
- OSCILLATION
- RELAXATION

Temperature Control Unit  
-150°C to 550°C



Controller System



- f 10<sup>-6</sup> to 150 Hz
- T 0.1µNm to 200mNm
- Torsional Load
- Static or Dynamic

Samples  
Φ - 10 to 25 mm  
h - 40 to 100 mm

## TEST METHOD

### BOHLIN RHEOMETER



- START UP OF RHEOMETER SYSTEM
- SELECT THE CORRECT MEASURING SYSTEM
- INSTALLING THE MEASURING SYSTEM

## TEST METHOD

### BOHLIN RHEOMETER

- SETTING THE CORRECT GAP
- SAMPLE LOADING


## TEST METHOD

### BOHLIN RHEOMETER



## TEST METHOD

### BOHLIN RHEOMETER



## TEST METHOD

### BOHLIN RHEOMETER

LOW STRAIN - 0.0065% (Linear Viscoelastic Properties)

HIGH STRAIN - 0.2% (Non Linear Viscoelastic Properties and Damage)

## TEST METHOD

### BOHLIN RHEOMETER

## TEST METHOD

### BOHLIN RHEOMETER

## TEST METHOD

### BOHLIN RHEOMETER

Low % Strain  
Linear Viscoelastic Properties

High % Strain  
Non Linear Viscoelastic  
Properties and Damage

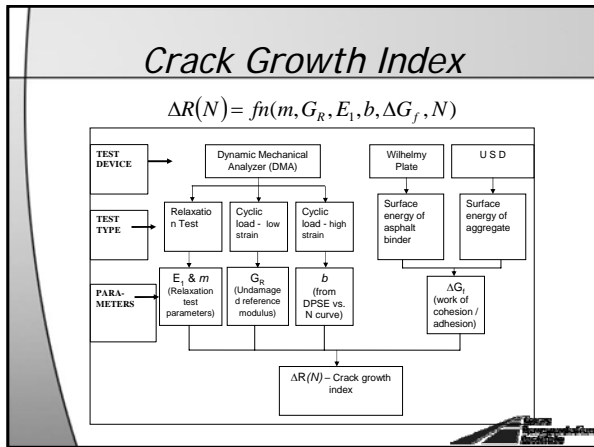
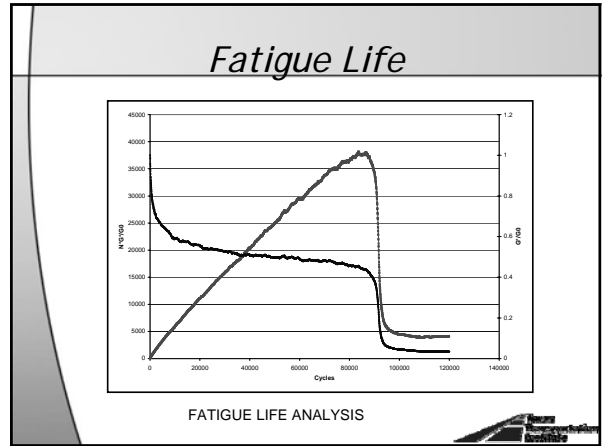
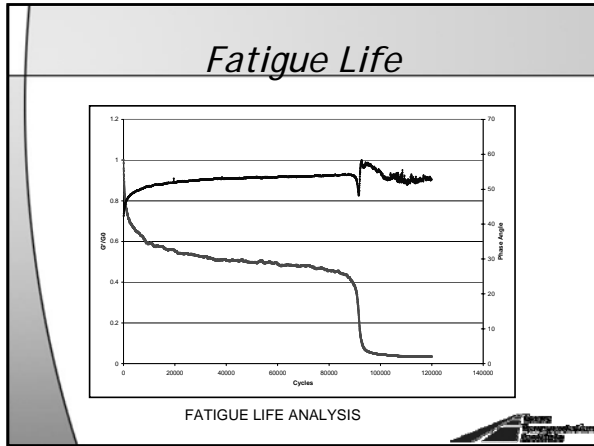
## Data Analysis

Two types:

- Direct estimation of fatigue life in terms of number of cycles to failure
  - Straightforward and simple
  - Higher variability
  - Gross measure of FAM performance
- Estimation of crack growth index
  - Based on fracture mechanics
  - Lower variability
  - Accounts for different material properties

## Fatigue Life

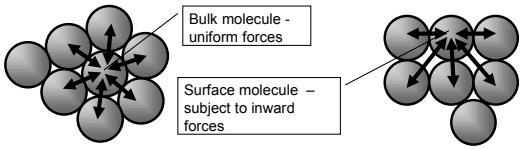
- Obtain summary raw data file from DMA
- Plot  $G^*$  vs. time or number of load cycles
- Plot  $\phi$  vs. time or number of load cycles
- Plot  $NxG^*(N)/G^*(1)$  vs. number of load cycles
- Obtain fatigue life





### Surface Energy

**Definition**  
Energy required to form a unit area of new surface

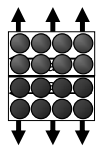


Bulk molecule - uniform forces  
Surface molecule - subject to inward forces

**Note**  
Interface is a special type of a surface

### Surface Energy

**Adhesion**

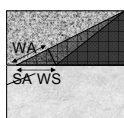


→ Surface energy of Interface =  $\gamma_{AS}$   
→ Work done for 'da' of material A =  $\gamma_A da$   
→ Work done for 'da' of material S =  $\gamma_S da$   
→ 'da' of interface A-S lost =  $\gamma_{AS} da$   
→ Adhesive bond energy  $\Delta G_{AS} = \gamma_A + \gamma_S - \gamma_{AS}$

Greater magnitude of  $\Delta G_{AS}$  → stronger the bond between the two materials

### Surface Energy

**Moisture Damage**



SA = lost aggregate-asphalt interface ( $-\gamma_{SA}$ )  
WS = new water-aggregate interface ( $\gamma_{WS}$ )  
WA = new water-asphalt interface ( $\gamma_{WA}$ )

Total energy used  
 $\Delta G_{WAS} = \gamma_{WA} + \gamma_{WS} - \gamma_{SA}$   
 $\leq 0$  !! (Typically)

Lesser magnitude of  $\Delta G_{WAS}$  → greater work required by external loads to drive water to strip bitumen from aggregate → lesser moisture sensitivity

### Surface Energy

Conditions favorable to resist moisture damage:

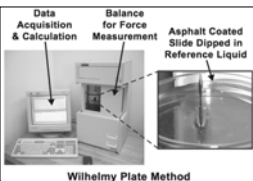
- (i) High value of adhesive bond energy  $\Delta G_{AS}$
- (ii) Low value of reduction in free energy of the system  $|\Delta G_{WAS}|$

Combining the above two statements:

$$\text{Resistance to moisture damage} \propto \frac{\Delta G_{AS}}{|\Delta G_{WAS}|} = ER$$

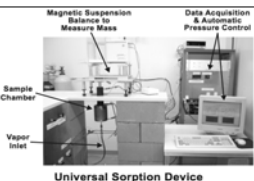
→  $f(\text{surface energy components of A, S, \& W})$

### Surface Energy



**Wilhelmy Plate Method**

Asphalt:  
Contact Angle Method



**Universal Sorption Device**

Aggregate:  
Vapor Adsorption Method

### Surface Energy

**Wilhelmy Plate Test**

Output: Contact angles

Analysis: Work of adhesion with probe liquids

Result: Three surface energy components

**Adsorption Test (USD)**

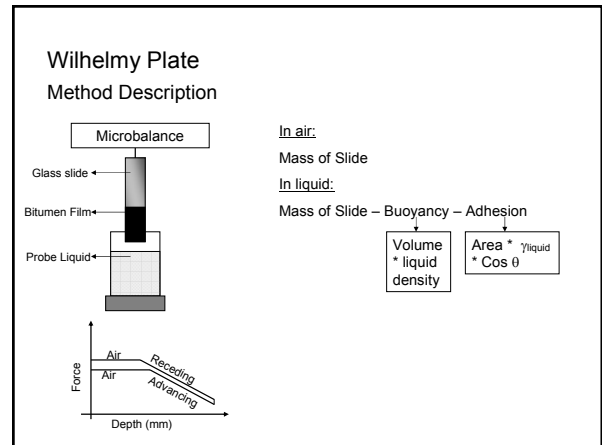
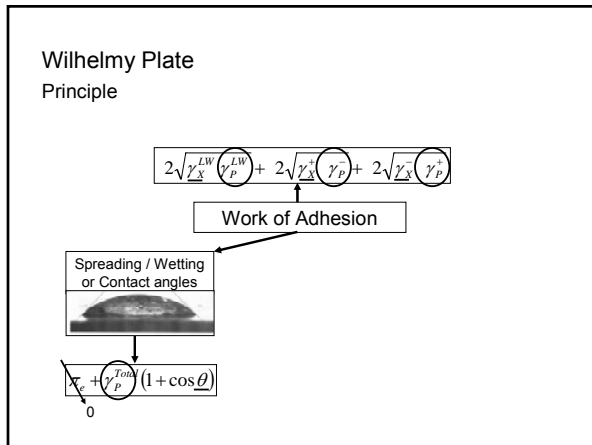
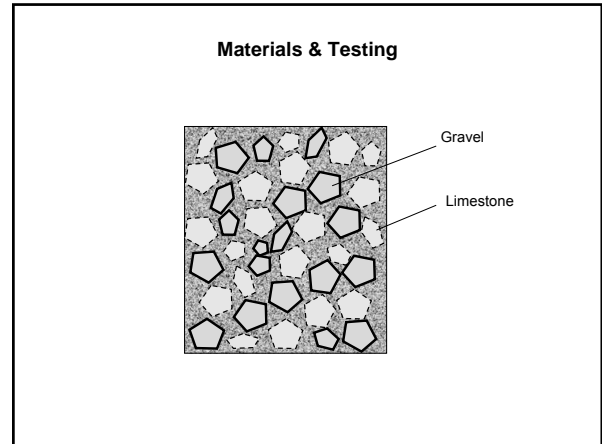
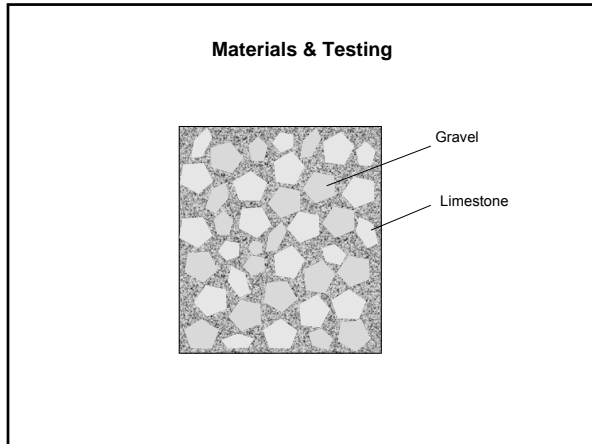
Output: Adsorption isotherm

Analysis: Work of adhesion with probe vapors

Result: Three surface energy components

Performance related parameters:  
(1)  $\Delta G_{AS}$  (2)  $\Delta G_{WAS}$

Moisture Sensitivity of Mixes:  
Field / Laboratory Performance



**Wilhelmy Plate Sampling**

Obtain a representative sample of the asphalt binder.

Approximately 50 g of asphalt binder stored in a small metallic container is required for this test.

**Wilhelmy Plate Preparation of Test Samples**

Heat the container with asphalt binder in an oven to the mixing temperature for about 1 hour and place it over a heating plate.

Set the temperature of the heating plate so that the asphalt binder remains at the mixing temperature.

Stir the liquid asphalt binder from time to time throughout the sample preparation process.

### Wilhelmy Plate

#### Preparation of Test Samples

Pass the end of the glass slide intended for coating six times on each side through the blue flame of a propane torch to remove any moisture.



### Wilhelmy Plate

#### Preparation of Test Samples

Dip the slide into the molten bitumen to a depth of approximately 15 mm.



### Wilhelmy Plate

#### Preparation of Test Samples

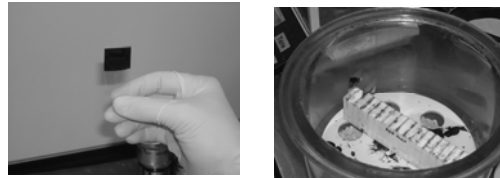
Drain excess binder is allowed to drain from the plate until a very thin (0.18 to 0.35 mm) and uniform layer at least 10 mm thick remains on the plate.

A thin coating is required to reduce variability of the results.

### Wilhelmy Plate

#### Preparation of Test Samples

A thin coating is required to reduce variability of the results. Turn the plate with the uncoated side downward and carefully place it in the slotted slide holder.



### Wilhelmy Plate

#### Preparation of Test Samples

If necessary, the heat-resistant slide holder with all the coated slides is placed in the oven after coating for 15 to 30 seconds to obtain the desired smoothness.

Place the binder-coated plates in a desiccator overnight.

### Wilhelmy Plate

#### Test Method

Ensure that the microbalance is calibrated in accordance with the manufacturer specifications prior to the start of test.

Remove one asphalt binder coated slide from the desiccator at a time.

Measure the width and thickness of the asphalt binder slide to an accuracy of 0.01 mm to calculate its perimeter. The measurements must be made just beyond 8 mm from the edge of the slide to avoid contamination of the portion of coating that will be immersed in the probe liquid.

### Wilhelmy Plate

#### Test Method

Suspend the glass slide coated with asphalt binder from the microbalance using a crocodile clip.

Ensure that the slide is horizontal with respect to the base of the balance.

Fill a clean glass beaker with the probe liquid to a depth of at least 10 mm and place it on the balance stage.

### Wilhelmy Plate

#### Test Method

Raise the stage manually to bring the top of the probe liquid in proximity to the bottom edge of the slide



### Wilhelmy Plate

#### Test Method

During the test, the stage is raised or lowered at the desired rate via a stepper motor controlled by the accompanying software.

A rate of 40 microns per second is recommended to achieve the quasi-static equilibrium conditions for contact angle measurement.

The depth to which the sample is immersed in the probe liquid is set to 8 mm.

### Wilhelmy Plate

#### Test Method

Five probe liquids are recommended for use with this test. These are water, ethylene glycol, methylene iodide (diiodomethane), glycerol, and formamide.

All reagents must be high-purity grade (>99%). Contact angles must be measured for at least three replicates with each probe liquid for each asphalt binder.

Since methylene iodide is a light-sensitive material, cover the beaker containing methylene iodide with black tape to reduce the effect of light.

### Wilhelmy Plate

#### Test Method

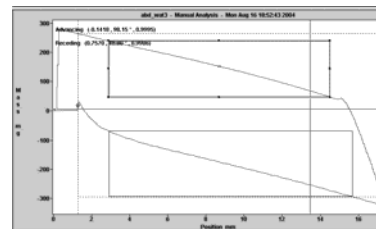
Dispose the probe liquid in the beaker after testing with three asphalt binder slides, and use a fresh sample of the probe liquid for each different type of binder. Store all probe liquids in air tight containers and do not use after prolonged exposure to air in open-mouthed beakers.

Tests must be completed within 24 to 36 hours from the time of preparation of the slides.

### Wilhelmy Plate

#### Results

Select a representative area of the line for regression analysis. The software reports the advancing & receding contact angles based on the area selected using the aforementioned equation.



### Wilhelmy Plate

#### Results

Report force measurements that are not smooth, i.e., if sawtooth-like force measurements are observed due to slip-stick behavior between the probe liquid and the asphalt binder

The typical standard deviation of the measured contact angle for each pair of liquid and asphalt binder based on measurements with three replicate slides is less than 2°.

### Wilhelmy Plate

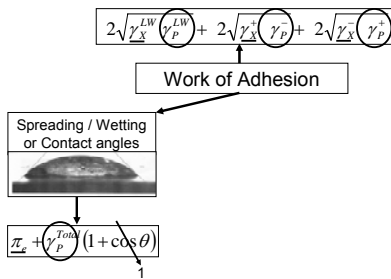
#### Results

The contact angle of each replicate and probe liquid is used with the surface energy analysis workbook that conducts the required analysis to determine the three surface energy components of the asphalt binder and the standard deviations of these components.

This workbook also verifies the accuracy and consistency of the measured contact angles and integrates data from other test methods such as the surface energy components of aggregates to determine various parameters of interest that are related to the performance of asphalt mixes.

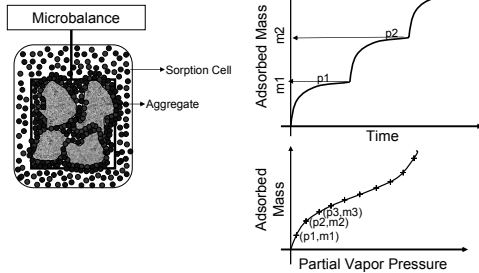
### U S D

#### Principle



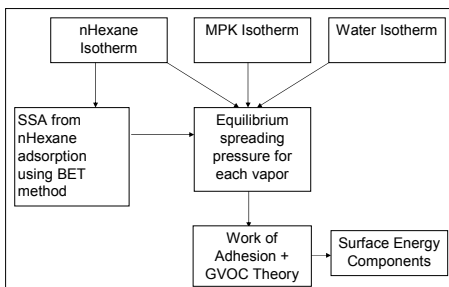
### U S D

#### Method Description



### U S D

#### Method Description



### U S D

#### Sampling

Sieve the sample to obtain about 100 g of aggregates passing a 4.75 mm sieve (#4) and retained on a 2.36 mm sieve (#8)

U S D

Preparation of Samples

Thoroughly wash about 25 g of the aggregate in a 2.36 mm sieve with deionized or distilled water.

The quality of water used for cleaning of aggregates must be comparable to the quality of water used for gas chromatography.

Place the clean aggregate sample in an oven at 150°C for 8 hours, and thereafter transfer it to a desiccator at room temperature for at least 8 hours before testing.

U S D

Test Method

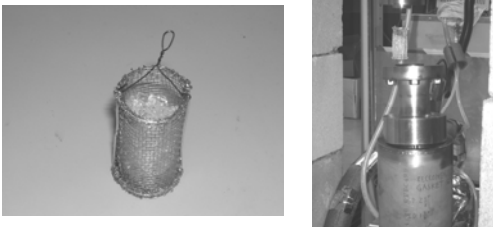
The samples are held in a wire mesh basket during the test.

Rinse the basket with acetone and air dry.

U S D

Test Method

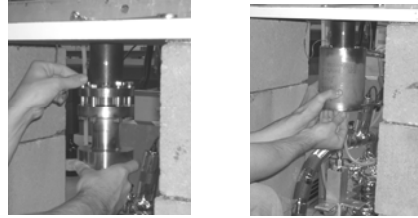
Transfer the aggregate sample to the basket and suspend the basket from the hook underneath the suspension balance.



U S D

Test Method

Seal the sorption cell with the coupling with the suspension balance using a viton O-ring. A metal jacket connected to a water bath is used around the sorption cell to maintain temperature.



U S D

Test Method

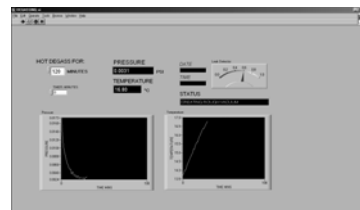
Activate and deactivate the magnetic suspension coupling repeatedly until stable and consistent readings are observed. This can also be automatically executed with the "Horizontal Centering" module of the SEMS software.



U S D

Test Method

The temperature and degassing times can be controlled manually or automatically using the "Degassing" module of the SEMS software.



## U S D

### Test Method

Degas the sample and the test manifold by using a mechanical vacuum pump (first 2 hours of degassing at 70°C followed by 25°C for another 4 hours).

The pressure in the cell must be maintained below 20 millitorr during the last 4 hours of degassing. Monitor the mass of the sample for the last 1 hour of the degassing time to ensure that it is stable. If the mass continues to decrease it indicates that the sample is still losing physically adsorbed particles from its surface and more degassing time is required.

## U S D

### Test Method

After completion of degassing isolate the vacuum pump from the adsorption system.

Monitor the pressure of the system for a few minutes to ensure that there is no significant leak.

Typically, a leak that allows the system pressure to increase by more than 40 millitorr per hour is unacceptable. In such cases, retighten and replace the seal with the sorption cell and repeat the degassing process.

## U S D

### Test Method

Activate the "Adsorption Test" module of the SEMS software to control and execute the adsorption test.



## U S D

### Test Method

Provide the necessary inputs to the software, such as volume of aggregate (computed by dividing the mass of the aggregate by its density) and probe vapor to execute the test.

Start the test from the SEMS software.

## U S D

### Test Method

A mechanical isolation valve is used between each of the probe liquid tanks and the system to prevent accidental exposure of the system to the probe vapors.

Open the valve corresponding to the probe vapor for the test.

Close this valve after completion of the test and before changing or degassing samples.

## U S D

### Test Method

Three probe vapors water, methyl propyl ketone (MPK), and hexane are recommended for this test.

All reagents must be high-purity grade (>99 percent).

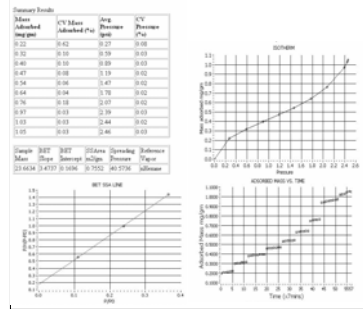
After the filling the respective liquid tanks in the manifold for the first time, degas the tanks to remove any air trapped during the process of refilling.

Typically, 100 ml of n-hexane lasts for approximately 15 tests, and 100 ml of MPK and water last for 60 tests.

U S D  
Results

At end of test the software reports a summary of final results that includes the adsorption isotherm, specific surface of the aggregate, and spreading pressure based on the specific surface area

U S D  
Results



U S D  
Results

The typical coefficient of variation (standard deviation / average) for the spreading pressure for each pair of probe vapor and aggregate based on three replicate measurements is about 15%.

U S D  
Results

Certain corrections must be applied in order to obtain the correct SSA and spreading pressures that can be combined to determine the three surface energy components.

Adsorption isotherms for each of the three probe vapors reported by SEMS are used with the [surface energy analysis workbook](#) that conducts the required analysis.



**APPENDIX B:**

**PROTOCOL TO MEASURE SPREADING PRESSURES  
AND METHOD TO DETERMINE SURFACE FREE ENERGY  
OF AGGREGATES USING THE SPREADSHEET CALCULATOR  
(PRODUCT 3: PART 1 OF 2)**



This appendix includes a draft protocol to determine the surface free energy of aggregates using the USD. The draft protocol is in AASHTO format and was originally developed during the NCHRP 9-37 project. The appendix also includes a guide to use Microsoft Excel<sup>®</sup> spreadsheets to compute the surface free energy components of the aggregates from the data generated using the USD software.

## **1. Scope**

- 1.1 This test method covers the procedures for preparing samples and measuring adsorption isotherms using a sorption device with an integrated Surface Energy Measurement System (SEMS) to determine the three surface energy components of asphalt binders.
- 1.2 This standard is applicable to aggregates that pass through 4.75 mm sieve (No. 4) and are retained on a 2.36 mm sieve (No. 8).
- 1.3 This method must be used in conjunction with the manual for mathematical analysis to determine surface energy components from spreading pressures or the computerized spreadsheets that were developed to carry out this analysis.
- 1.4 *This standard may involve hazardous material, operations, and equipment. This standard is not intended to address all 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 its use.*

## **2. Referenced Documents**

- 2.1 AASHTO Standards T 2 Practice for sampling aggregates

### 3. Definitions

- 3.1 *Surface Energy*-  $\gamma$ , or surface free energy of a material is the amount of work required to create unit area of the material in vacuum. The total surface energy of a material is divided into three components namely the Lifhsitz-van der Waals component, the acid component, and the base component.
- 3.2 *Equilibrium spreading pressure* -  $\pi_e$ , is the reduction in surface energy of the solid due to adsorption of vapors at its saturation vapor pressure on the surface of the solid.
- 3.3 *Probe Vapor*, within the context of this test, refers to vapors from any of the pure, homogeneous liquids that do not chemically react or dissolve with aggregates and is used to measure the spreading pressure with the aggregate. The three surface energy components of the probe vapor must be known at the test temperature from the literature.
- 3.4 *Relative Vapor Pressure*, within the context of this test, refers to the ratio of the pressure of the vapor to its saturation vapor pressure and can vary from 0 (complete vacuum) to 1 (saturation vapor pressure).
- 3.5 *Adsorption Isotherm*, of a vapor with an aggregate, is the relationship between the equilibrium mass of vapor adsorbed per unit mass of the aggregate and the relative vapor pressure of the vapors at a constant temperature.

### 4. Summary of Method

- 4.1 Clean aggregate samples are degassed under high temperature and vacuum in an airtight sorption cell. Vapors of probe liquids are introduced into the sorption cell in controlled and gradually incremental quantities to achieve different relative pressures. The equilibrium mass of the vapor adsorbed to the solid surface is recorded for each relative pressure to obtain the adsorption isotherm. The adsorption isotherm is used to compute the equilibrium spreading pressure of the probe vapor with the aggregate.

- 4.2 Equilibrium spreading pressure with different probe vapors are used with equations of work of adhesion to determine the three surface energy components of the aggregate.

## **5. Significance and Use**

- 5.1 Surface energy components of aggregates is an important material property that is related to the performance of hot mix asphalt. Surface energy components of aggregates can be combined with the surface energy components of asphalt binders to quantify the work of adhesion between these two materials and the propensity for water to displace the asphalt binder from the asphalt binder-aggregate interface. These two quantities are related to adhesive fracture properties and moisture sensitivity of the asphalt mix.

## **6. Apparatus**

- 6.1 A sorption device integrated with the SEMS comprising of an air tight adsorption cell, a magnetic suspension balance that measures the mass the sample in the sorption cell in non-contact mode, a manifold with vacuum pump, temperature control, probe liquid containers with appropriate valves and controls to regulate the flow of vapors into the sorption cell, and associated software for test control and analysis. The micro balance must have a least count of 10  $\mu$ gm with a capacity to weight at least 50 gm.
- 6.2 Temperature of the sorption cell, piping that carry vapors, and the buffer tank are maintained using a water bath that is automatically controlled by the SEMS software.
- 6.3 An oven capable to heating up to 150°C is required to prepare aggregate samples before testing.

## **7. Sampling**

- 7.1 Obtain a representative sample of the aggregate according to procedure AASHTO T 2. Sieve the sample to obtain about 100 gm of aggregates passing 4.75 mm sieve (No. 4) and retained on 2.36 mm sieve (No. 8).

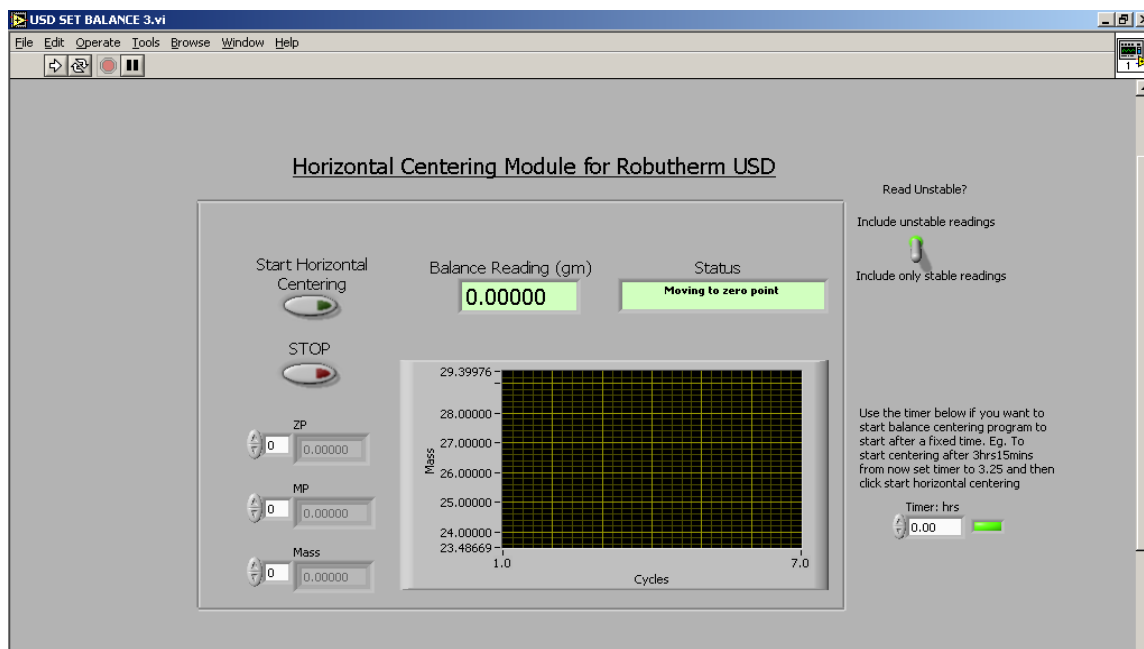
## **8. Preparation of Test Samples**

- 8.1 Thoroughly wash about 25 gm of the aggregate in a 2.36 mm sieve with deionized or distilled water. Place the clean aggregate sample in an oven at 150°C for 8 hours, and thereafter transfer it to a desiccator at room temperature for at least 8 hours before testing.

## **9. Procedure**

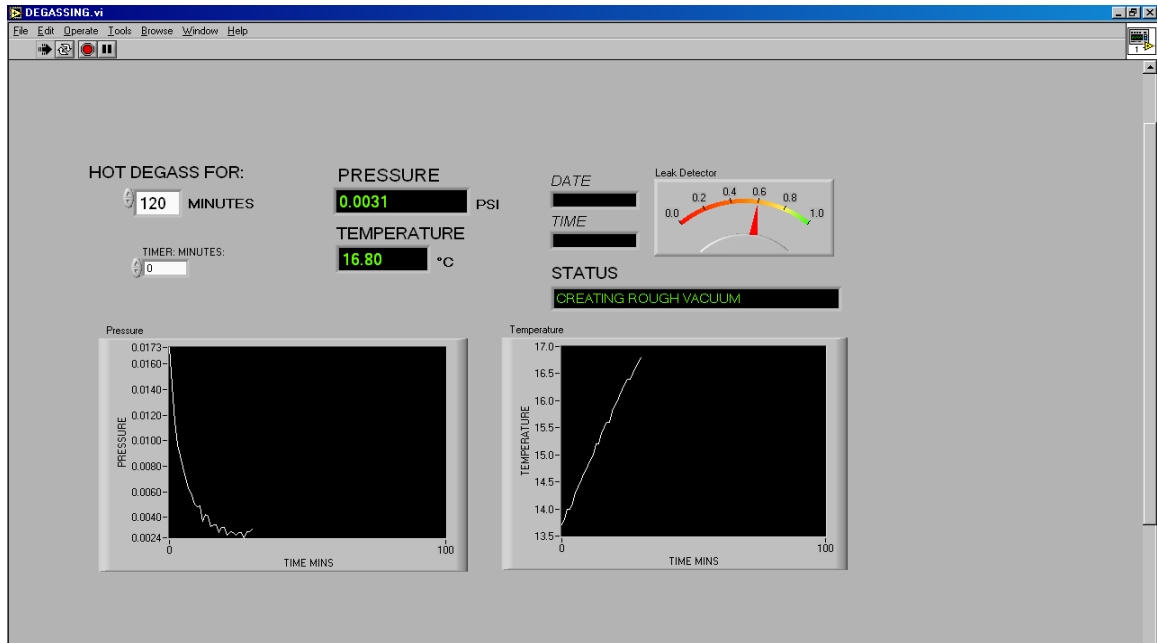
- 9.1 The samples are held in a wire mesh basket during the test. Rinse the basket with acetone and air dry it. Transfer the aggregate sample to the basket, and suspend the basket from the hook underneath the suspension balance. Seal the sorption cell with the coupling with the suspension balance using a viton O-ring. A metal jacket connected to a water bath is used around the sorption cell to maintain temperature.
- 9.2 In order to obtain stable and consistent readings with the magnetic suspension balance, it is necessary that the sample basket and magnetic suspension coupling are in vertical and horizontal alignment with each other. Activate and deactivate the magnetic suspension coupling repeatedly until the stable and consistent readings are observed. This is an indication that the balance is aligned. This process, referred to as centering of balance, and can also be automatically executed with the “Horizontal Centering” module of the SEMS software (Figure B.1).
- 9.3 Degass the sample and the test manifold by drawing vacuum from the system using a mechanical vacuum pump. After the first 2 hours of degassing at 70°C,

reduce the temperature of the manifold to 25°C (test temperature) and continue degassing for another four hours. The pressure in the cell must be maintained below 20 millitorr during the last four hours of degassing. The temperature and degassing times can be controlled manually or automatically using the “Degassing” module of the SEMS software (Figure B.2). Monitor the mass of the sample for the last one hour of the degassing time to ensure that it is stable. If the mass continues to show a decrease, it indicates that the sample is still losing physically adsorbed particles from its surface and more degassing time is required.



**Figure B.1. Auto Centering Module in SEMS Software.**

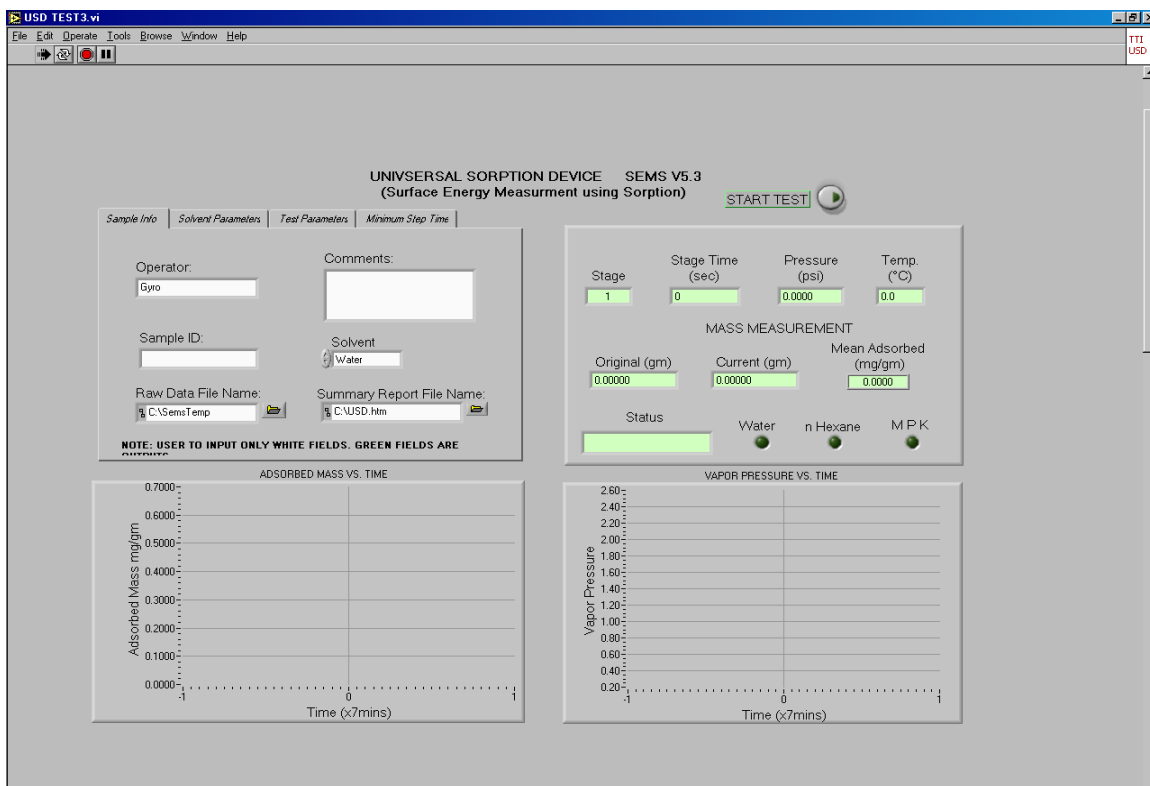
9.4 After completion of degassing, isolate the vacuum pump from the adsorption system. Monitor the pressure of the system for a few minutes to ensure that there is no significant leak. Typically, a leak that allows the system pressure to increase by more than 40 millitorr per hour is unacceptable. In such cases, the seal with the sorption cell must be retightened or replaced and the degassing repeated.



**Figure B.2. Degassing Module in SEMS Software.**

9.5 Activate the “Adsorption Test” module of SEMS software to control and execute the adsorption test (Figure B.3). Provide the necessary inputs to the software, such as volume of aggregate (computed by dividing the mass of the aggregate by its density) and probe vapor, to execute the test. Other inputs such as name and description of the sample, name and location of the summary and raw data file for saving results, minimum equilibrium time for each increment of relative pressure are also required. The minimum time of 15 minutes for equilibrium of each increment is recommended. Start the test from the SEMS software. A mechanical isolation valve is used between each of the probe liquid tanks and the system to prevent accidental exposure of the system to the probe vapors. Open the valve corresponding to the probe vapor for the test. Close this valve after completion of the test and before changing or degassing samples.





**Figure B.3. Adsorption Test Module in SEMS Software.**

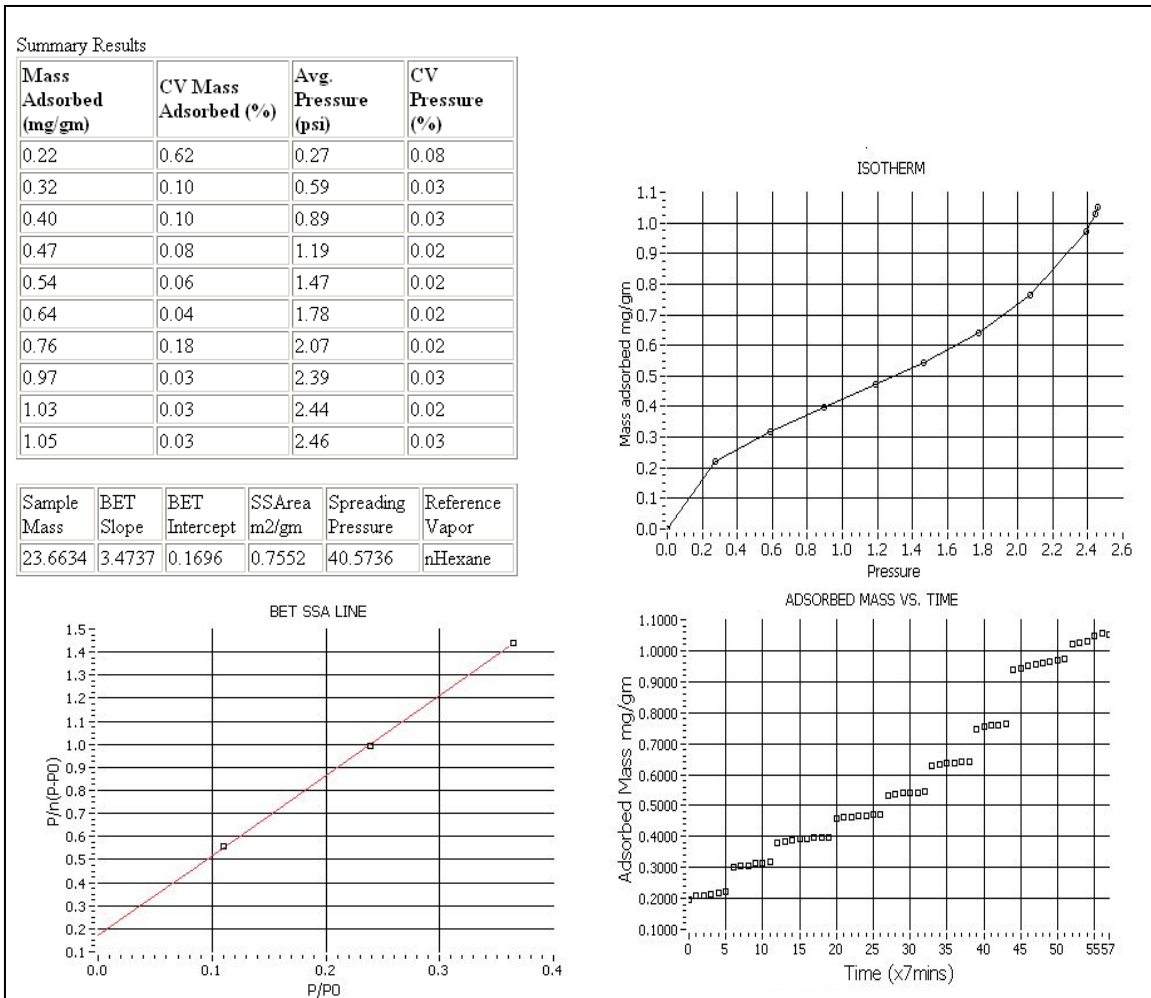
9.6 The test is controlled, and data are acquired using the SEMS software. The software regulates valves to dose probe vapors into the system in ten steps to achieve an increment of 0.1 in the relative pressure with each step. The mass of the sample is continuously acquired during this process by the SEMS software. The software computes the mass of vapor adsorbed in real time as the difference in the mass of sample at any time with the mass of the sample in vacuum after applying for corrections due to buoyancy. The software also corrects for any drift in the measurements due to the magnetic suspension coupling. Each increment of relative pressure is applied by the software after the mass of the sample comes into equilibrium due to adsorption of vapors from the previous increment or after the minimum time for equilibrium is achieved, whichever is later. The test is complete after the saturation vapor

pressure of the probe liquid is achieved in ten increments, and the equilibrium mass of vapor adsorbed is recorded for each increment.

- 9.7 Three probe vapors are recommended for this test. These are water, methyl propyl ketone (MPK), and hexane. All reagents must be high purity grade (>99 percent). After the liquids are filled in their respective tanks in the manifold for the first time, the liquid tanks must be degassed to remove any trapped air during the process of refilling. Typically, 100 ml of n-Hexane lasts for approximately 15 tests, and 100 ml of MPK and water last for 60 tests.

## **10. Calculations**

- 10.1 After completion of all ten increments in vapor pressure, the software reports a summary of final results that include the adsorption isotherm, specific surface of the aggregate with BET equations, and spreading pressure based on the specific surface area and the adsorption isotherm (Figure B.4).
- 10.2 Although the SEMS software reports specific surface areas and spreading pressures for each test, certain corrections must be applied in order to obtain the correct specific surface area and spreading pressures that can be combined to determine the three surface energy components. Therefore, the adsorption isotherms with each of the three probe vapors reported by SEMS are used with the surface energy analysis workbook that conducts the required analysis to determine the specific surface area and the three surface energy components of the aggregate and the standard deviations of these components. This user-friendly workbook also integrates data from other tests such as the surface energy components of asphalt binders to determine various parameters of interest that are related to the performance of asphalt mixes.



**Figure B.4. Results Reported by SEM5 Software.**

## 11. Procedure to Use the Accompanying Spread Sheet for Calculations

The SPREADING PRESSURE TO SE.xls workbook contains five main worksheets: Spreading Pressures, Matrix Analyzer, Adh Coh by Weight, Adh Coh by Area, Summarized USD Results, and Aggregate Input Sheets. The Aggregate Input Sheets have the yellow tabs in Figure B.5. The name of the Aggregate Input Sheet can be whatever the user chooses as long as it is accepted by Excel. The layout of the Aggregate Input Sheet is displayed in Figure B.6 below. This layout is repeated to the right for the other two probe liquids. The procedure for entering data is as follows:



**Figure B.5. Worksheets Contained in SPREADING PRESSURE TO SE.**

1. Click on the Aggregate Input Sheet that data will be entered in. If none are available, right click on one of the Aggregate Input Sheets, select “Move or Copy...”, check the “Create a copy” box and choose the location for the sheet. Once the new sheet has been created, it can be renamed as needed.
2. Click on “Import MPK #1” or the respective button for the data needing to be input.
3. An “Open” window will appear. Choose the appropriate data file to open. The software on the USD outputs the test data in .html format; therefore, the programming in this spreadsheet only recognizes that file type.
4. Open all files for the aggregate.
5. Once all files have been opened, it is time to check the calculation for specific surface area (SSA) of the aggregate. The SSA of the aggregate is based off the information from the probe liquid n-Hexane. Figure B.7 illustrates the setup of the SSA information in the worksheet. The SSA for each replicate is displayed in the table at the top. If one of the replicates has a large variation from the others it can be removed by selecting “No” instead of “Yes” under the Include column.
6. All the data have been entered.
7. Click on the “Summarized USD Results” tab.
8. On the left-hand side of the screen is a column of green cells as shown in Figure B.8 below. Enter the name of the Aggregate Input Sheet in one of the green cells. It must be labeled identical or the spreadsheet will not recognize it. If there are no available spaces, copy as many rows as necessary and paste it right below the main table. All the surface energy information will appear as soon as the name of the Aggregate Input Sheet is entered.

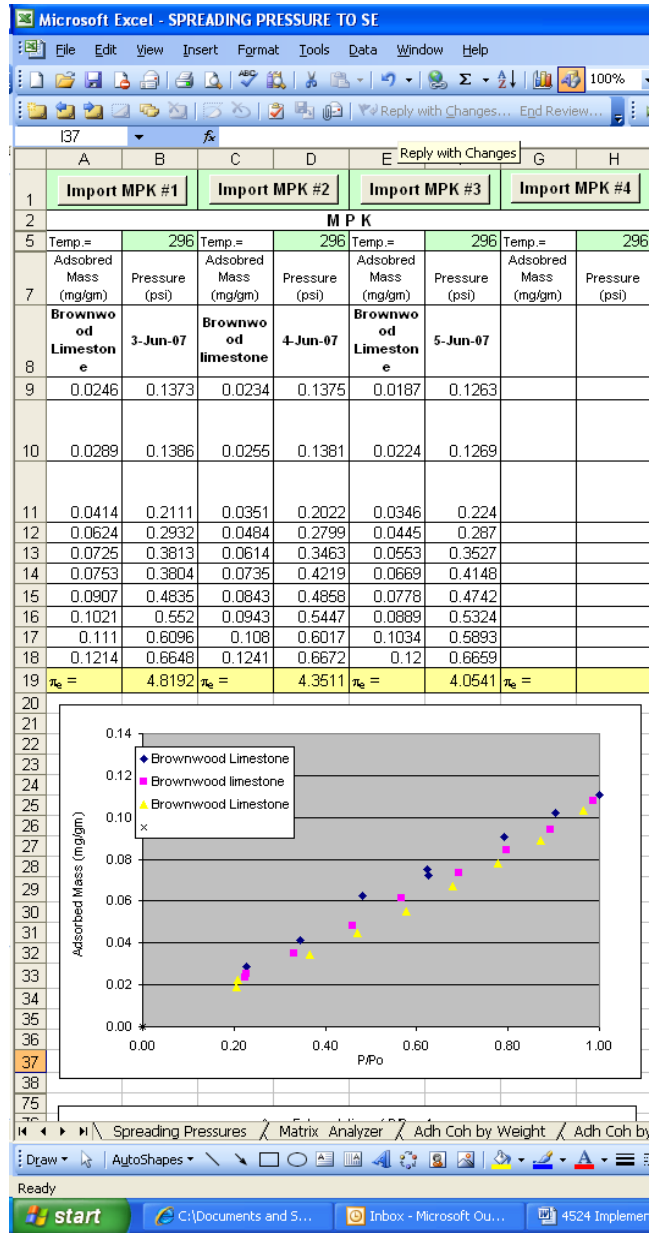
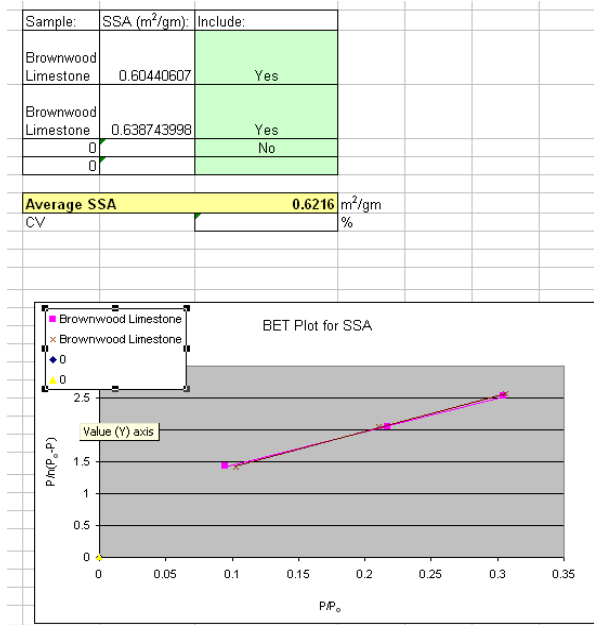


Figure B.6. Aggregate Input Sheet Layout.



**Figure B.7. Managing SSA.**

		Specific Surface Areas (sqm/gm)						Spreading Pressure MPK (e)			
		1	2	3	4	Average	CV %	1	2	3	4
4	Brownwood	0.60	0.64			0.62	3.91	4.82	4.35	4.05	
5	Knippa	0.25	0.29			0.27	9.97	9.79	9.12		
6	Odessa	0.78	0.80	0.64		0.74	11.83	6.21	7.55		
7	Type D	0.57	0.50			0.54	9.86	7.48	7.12		
8	US 281	0.44	0.35	0.31		0.37	18.60	6.49	5.82	5.44	
9	Diabase	0.42	0.32	0.31		0.35	17.83	7.46	8.38	7.08	
10	I 30	0.71	0.59	0.60		0.64	10.58	4.08	3.57	3.90	
11	Brownwood New	1.01	1.01			1.01	0.15	2.59	2.59	2.64	2.81
12	Fordyce River Gravel - New	0.45	0.45	0.50		0.47	6.36	1.57	5.64	5.19	
13	Knippa Traprock - New	1.25	1.00	0.64		0.96	31.74	2.73	2.81	2.81	
14	Mill Creek Granite - New	0.53	0.40			0.47	19.27		4.85	4.20	4.13
15	MM Sandstone - New	0.79	1.02	1.00		0.94	13.52		3.07	2.77	2.96
16	RB Granite	0.49	0.51	0.56		0.52	6.83	47.26	54.84	40.33	
17	RC Limestone	1.03	0.79	0.87	1.31	1.00	23.32	35.75	37.16	29.17	
18											
19	<b>Material Names Above Must Match Worksheet Names</b>										

**Figure B.8. Summarized USD Results.**

**APPENDIX C:**

**PROTOCOL TO MEASURE CONTACT ANGLE AND METHOD  
TO DETERMINE SURFACE FREE ENERGY OF ASPHALT  
BINDERS USING THE SPREADSHEET CALCULATOR  
(PRODUCT 3: PART 2 OF 2)**





This appendix includes a draft protocol to determine the surface free energy of asphalt binders using the Wilhelmy plate device. The draft protocol is in AASHTO format and was originally developed during the NCHRP 9-37 project. The appendix also includes a guide to use Microsoft Excel<sup>®</sup> spreadsheets to compute the surface free energy components of the asphalt binders from the data generated from the Wilhelmy plate device.

## **1. Scope**

- 1.1 This test method covers the procedures for preparing samples and measuring contact angles using the Wilhelmy plate device to determine the three surface energy components of asphalt binders.
- 1.2 This standard is applicable to asphalt binders that do not contain particulate additives such as crumb rubber.
- 1.3 This method must be used in conjunction with the manual for mathematical analysis to determine surface energy components from contact angle measurements or the computerized spread sheets that were developed to carry out this analysis.
- 1.4 *This standard may involve hazardous material, operations, and equipment. This standard is not intended to address all 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 its use.*

## **2. Referenced Documents**

- 2.1 AASHTO Standards T40 Sampling of Bituminous Materials

### 3. Definitions

- 3.1 *Surface Energy*-  $\gamma$ , or surface free energy of a material, is the amount of work required to create unit area of the material in vacuum. The total surface energy of a material is divided into three components namely the Lifhsitz-van der Waals component, the acid component, and the base component.
- 3.2 *Contact Angle* -  $\theta$ , refers to the equilibrium contact angle of a liquid on a solid surface measured at the point of contact of the liquid-vapor interface with the solid.
- 3.3 *Advancing Contact Angle*, within the context of this test, refers to the contact angle of a liquid with the solid surface as the solid surface is being immersed into the liquid.
- 3.4 *Receding Contact Angle*, within the context of this test, refers to the contact angle of a liquid with the solid surface as the solid surface is being withdrawn from the liquid.
- 3.5 *Probe Liquid*, within the context of this test, refers to any of the pure, homogeneous liquids that does not react chemically or dissolve with asphalt binders and is used to measure the contact angles with the binder. The three surface energy components of the probe liquid must be known at the test temperature from the literature.
- 3.6 *Mixing Temperature*, within the context of this test, refers to the temperature at which the viscosity of the asphalt binder is approximately 0.170 Pas, or any other temperature that is prescribed or determined by the user for use as the mixing temperature with aggregates to prepare hot mix asphalt.

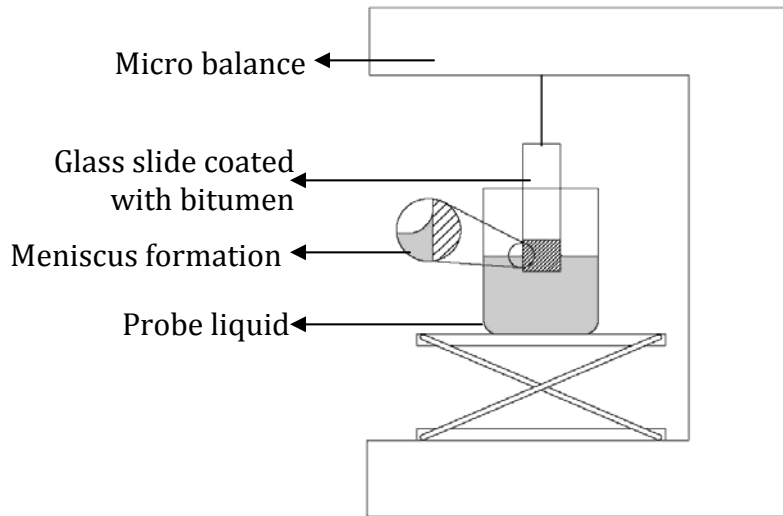
### 4. Summary of Method

- 4.1 A glass slide coated with the asphalt binder and suspended from a micro balance is immersed in a probe liquid. From simple force equilibrium conditions the contact angle of the probe liquid with the surface of the asphalt

binder can be determined. The analysis to obtain the contact angle is carried out using a software accompanying the device.

4.2 Contact angles measured with different probe liquids are used with equations of work of adhesion to determine the three surface energy components of the asphalt binder.

4.3 Figure C.1 presents a schematic of the Wilhelmy plate device.



**Figure C.1. Schematic of the Wilhelmy Plate Device.**

## **5. Significance and Use**

5.1 Surface energy components of asphalt binders is an important material property that is related to the performance of hot mix asphalt. Surface energy components of asphalt binders can be used to determine the total surface energy and cohesive bond strength of this material. The cohesive bond strength of asphalt binders is related to the work required for micro cracks to propagate within the asphalt binder in an asphalt mix, which is related to the fatigue cracking characteristics of the mix.

- 5.2 Surface energy components of asphalt binders can also be combined with the surface energy components of aggregates to compute the work of adhesion between these two materials and the propensity for water to displace the asphalt binder from the asphalt binder-aggregate interface. These two quantities are related to the moisture sensitivity of the asphalt mix.

## **6. Apparatus**

- 6.1 Wilhelmy plate device – This device comprises of a micro balance with a motor controlled stage that can be raised or lowered at desired speed to immerse a slide with asphalt binder in the probe liquid in advancing mode and to withdraw the slide from the probe liquid in receding mode.
- 6.2 A data acquisition and analysis software to collect the data and determine the contact angles.
- 6.3 An oven capable of heating up to 150°C is required to heat asphalt binders for sample preparation. Microscope glass slides (24 mm x 60 mm No. 1.5) are required to serve as substrates for the asphalt binder and a vernier calipers to measure the dimensions of the slide. A heating plate with temperature control is required for maintaining the temperature of the asphalt binder during the sample preparation process.
- 6.4 The tests are conducted at 25°C. If the room temperature is significantly different from the test temperature, then an appropriate environmental chamber may be required to house the apparatus.
- 6.5 A slotted slide holder is required to hold the finished asphalt binder slides.

## **7. Sampling**

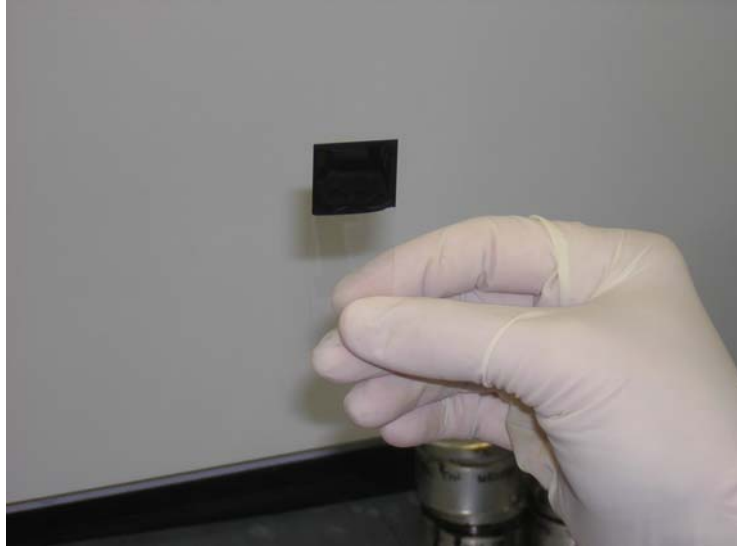
- 7.1 Obtain a representative sample of the asphalt binder according to procedure AASHTO T 40. Approximately 50 gm of asphalt binder stored in a small metallic container will be required for this test.

## **8. Preparation of Test Samples**

- 8.1 Heat the container with asphalt binder in an oven to the mixing temperature for about one hour and place it over a heating plate. Set the temperature of the heating plate so that the asphalt binder remains at the mixing temperature. The liquid asphalt binder is stirred from time to time throughout the sample preparation process.
- 8.2 Pass the end of the glass slide intended for coating six times on each side through the blue flame of a propane torch to remove any moisture. Dip the slide into the molten bitumen to a depth of approximately 15 mm. Excess bitumen is allowed to drain from the plate until a very thin (0.18 to 0.35 mm) and uniform layer of at least 10 mm remains on the plate. A thin coating is required to reduce variability of the results. Turn the plate with the uncoated side downwards (Figure C.2) and carefully place it in the slotted slide holder. If necessary, the heat-resistant slide holder, with all the coated slides is placed in the oven after coating for 15 to 30 seconds to obtain the desired smoothness. Place the bitumen-coated plates in a desiccator overnight.

## **9. Procedure**

- 9.1 The user must ensure that the micro balance is calibrated in accordance with the manufacturers specifications prior to the start of test.
- 9.2 One bitumen-coated slide is removed from the desiccator at a time. Measure the width and thickness of the asphalt binder slide to an accuracy of 0.01 mm to calculate its perimeter. The measurements must be made just beyond 8 mm from the edge of the slide to avoid contamination of the portion of coating that will be immersed in the probe liquid.



**Figure C.2. Glass Slide Coated with Asphalt Binder for Testing with the Wilhelmy Plate Device.**

- 9.3 Suspend the glass slide coated with asphalt binder the micro balance using a crocodile clip. Ensure that the slide is horizontal with respect to the base of the balance. Fill a clean glass beaker with the probe liquid to a depth of at least 10 mm and place it on the balance stage. Raise the stage manually to bring the top of the probe liquid in proximity to the bottom edge of the slide (Figure C.3).
- 9.4 During the test, the stage is raised or lowered at the desired rate via a stepper motor controlled by the accompanying software. A rate of 40 microns per second is recommended to achieve the quasi-static equilibrium conditions for contact angle measurement. Set the depth to which the sample is immersed in the probe liquid is set to 8 mm. Larger depths up to 15 mm may be used if the thickness of asphalt coating on the slide is uniform. The weight of the slide measured by the micro balance is recorded continuously during the advancing (stage is raised to dip the slide) and receding (stage is lowered to retract the slide from the liquid) process.
- 9.5 Five probe liquids are recommended for this test. These are water, ethylene glycol, methylene iodide (diiodomethane), glycerol, and formamide. All

reagents must be high purity grade (>99%). Contact angles must be measured for at least three replicates with each probe liquid for each asphalt binder.

- 9.6 Since methylene iodide is a light sensitive material, the beaker containing methylene iodide is covered with black tape to reduce the effect of light.
- 9.7 Dispose the probe liquid in the beaker after testing with three asphalt binder slides, and use a fresh sample of the probe liquid for each different type of binder. All probe liquids must be stored in air tight containers and must not be used after prolonged exposure to air in open-mouthed beakers.



**Figure C.3. Asphalt Binder Sample Suspended from Micro Balance for Immersion in Probe Liquid.**

## **10. Calculations**

- 10.1 From simple force equilibrium considerations, the difference between weight of a plate measured in air and partially submerged in a probe liquid, ( $\Delta F$ ), is expressed in terms of buoyancy of the liquid, liquid surface energy, contact

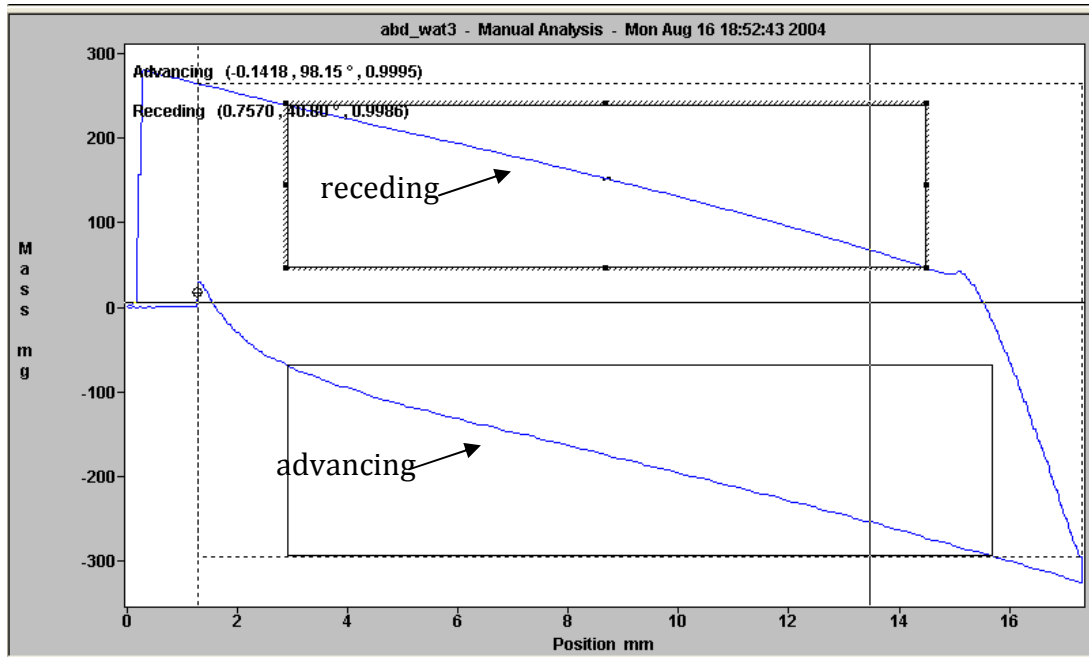
angle, and geometry of the plate. The contact angle between the liquid and surface of the plate is calculated from this equilibrium as:

$$\cos \theta = \frac{\Delta F + V_{im}(\rho_L - \rho_{air}g)}{P_t \gamma_L^{Tot}}$$

where  $P_t$  is the perimeter of the bitumen coated plate,  $\gamma_L^{Tot}$  is the total surface energy of the liquid,  $\theta$  is the dynamic contact angle between the bitumen and the liquid,  $V_{im}$  is the volume immersed in the liquid,  $\rho_L$  is the density of the liquid,  $\rho_{air}$  is the air density, and  $g$  the local acceleration due to gravitation. The accompanying software requires the density of the liquid, total surface tension of the liquid, dimensions of the sample, and local acceleration due to gravity as inputs to compute the contact angle using the force measurements from the micro balance.

- 10.2 Buoyancy correction based on slide dimensions and liquid density can introduce unwanted variability into the resulting contact angles. To eliminate these effects, the accompanying software performs a regression analysis of the buoyancy line and extrapolates the force to zero depth. The user must select a representative area of the line for regression analysis (Figure C.4). The software reports the advancing and receding contact angles based on the area selected using the aforementioned equation.
- 10.3 If the force measurements are not smooth, i.e., if saw tooth like force measurements are observed due to slip-stick behavior between the probe liquid and the asphalt binder, then report this along with the advancing and receding contact angles.
- 10.4 The contact angle of each replicate and probe liquid is used with the surface energy analysis workbook that conducts the required analysis to determine the three surface energy components of the asphalt binder and the standard deviations in these components. This workbook also verifies the accuracy and consistency of the measured contact angles and integrates data from other test methods such as the surface energy components of aggregates to determine various parameters of interest that are related to the performance of asphalt mixes.





**Figure C.4. Selection of Representative Area to Determine Advancing and Receding Contact Angles.**

## 11. Procedure to Use the Accompanying Spread Sheet for Calculations

The CONTACT ANGLE TO SE.xls spreadsheet contains two worksheets: Contact Angles and Summary Results. All of the information needed to compute the surface energy components is entered in the Contact Angle worksheet. There are three main steps to complete in the spreadsheet to compute the surface energy of asphalt binders

- 11.1 The first step is to enter the asphalt name, name of probe liquid, and corresponding contact angles obtained from each probe liquid in the Contact Angle workbook. Figure C.5 displays the areas (yellow) where information has to be entered into the spreadsheet. If there are no available spaces to enter a new asphalt binder, one can easily be created by copying the entire row from the asphalt to the grey line (including both) and pasting it below the bottom asphalt.

- 11.1.1 The name of the asphalt binder is entered into the top left-hand yellow highlighted cell. Do not number the asphalt in this cell, only label.
- 11.1.2. The probe liquids are located in the cells below the asphalt name. Every cell contains a drop-down list of all available probe liquids, which is linked to their corresponding material properties. Select the probe liquids used during the test and choose “None” for the remaining liquid cells.
- 11.1.3. The contact angles obtained from the Wilhelmy Plate are entered into the yellow cells in columns G, H, I, and J and on the row corresponding to the probe liquid used. Looking at Figure C.5, three replicates were run using formamide with resulting contact angles of 85.64, 86.43, and 86.09. Columns K and M display the average contact angle and standard deviation, respectively.

B9		04 MethyleneIodide							
A	B	F	G	H	I	J	K	M	
1	No.	NAME	Total SE of Liquid	$\theta_1$	$\theta_2$	$\theta_3$	$\theta_4$	Avg. $\theta$	$\alpha_\theta$
4	1	Lion 64-22 B							
5		01 Water	72.8	102.09	102.85	103.38	102.26	102.65	0.29
6		02 Glycerol	64.0	92.82	92.11	93.41	93.32	92.92	0.30
7		05 EthyleneGlycol	48.0	78.48	78.07	77.91		78.15	0.17
8		03 Formamide	58.0	85.64	86.43	86.09		86.05	0.23
9		04 MethyleneIodide	50.8	68.24	73.40	69.31	73.78	71.18	1.41
10		00 None	0.0					0.00	0.00
11		01 Water	0.0					0.00	0.00
12		02 Glycerol	0.0					0.00	0.00
13		03 Formamide	0.0					0.00	0.00
14		04 MethyleneIodide	0.0					0.00	0.00
15		05 EthyleneGlycol	0.0					0.00	0.00
16		06 Methylpropylketone	0.0					0.00	0.00
17		07 Hexane	0.0					0.00	0.00

**Figure C.5. Entering Contact Angle Information to Compute Surface Energy.**

11.2 The second step is computing the surface energy components. Once all the contact angle information has been entered, the surface energy can easily be computed. The sample number is located in column A of the spreadsheet as displayed in Figure C.5 above. This number is entered in the yellow cell in column AO as shown in Figure C.6. To the right of this is a button labeled

“Compute SE Components.” Click this button and the program will compute the LW, Acid, and Base components as well as the standard deviation of each.

	M	AK	AL	AM	AN	AO	AP	AQ	AR
1	$\sigma_0$	USER	CALCULATED	Select Asphalt # :	1	Compute SE Components	Plot Criteria		
3									
4									
5	0.29	Com.	SE	$\sigma$					
6	0.30	LW	18.5	0.9					
7	0.17	ACID	0.3	0.1					
8	0.23	BASE	1.2	0.3					
9	1.41	SUM OF SQAURES OF ERROR							
10	0.00	140							
11	0.00								
12	0.00								
13	0.00								
14	0.00								

Figure C.6. Computing Surface Energy Components.

11.3 The final step is to generate a summary table of the surface energy components of the tested asphalts. A summary table of the tested asphalts will be already be partly completed and located in the “Summary Results” worksheet. Do not delete this table. This table is directly linked to the “Contact Angles” worksheet and will automatically fill with results computed in that worksheet. If the table is too small, highlight the bottom row of cells and fill down as many rows as necessary as displayed in Figure C.7. This will incorporate all asphalts added under the “Contact Angles” worksheet. If the table is too large, highlight the unnecessary rows and delete them.

	A	F	G	H	I	J	K	L	M
1			Surface Energy Components				Standard Deviation		
2	No.	Asphalt	LW	Acid	Base	Total	sigma LW	Acid	Base
3	1	Lion 64-22 B	18.5	0.3	1.2	19.7	0.9	0.1	0.3
4	2	Lion 70-22 S	22.3	0.1	1.3	22.9	0.5	0.0	0.2
5	3	Lion 76-22 S	23.0	0.1	1.1	23.5	0.7	0.0	0.3
6									
7									
8									
9									
10									
11									
12									

Figure C.7. Summary Table of Results.



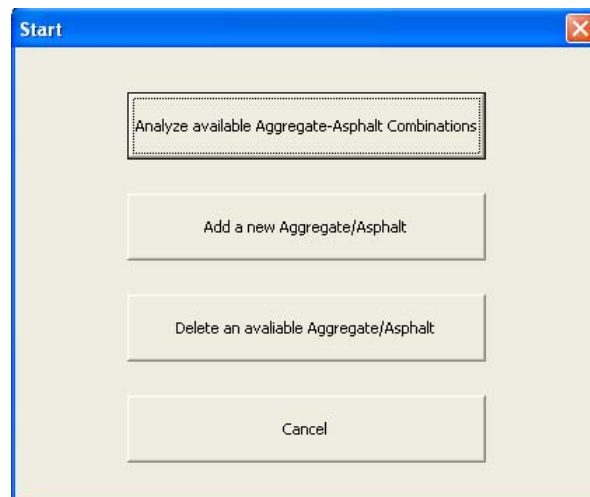
**APPENDIX D:**

**PROCEDURE TO USE AND POPULATE  
THE DATABASE OF SURFACE FREE ENERGIES  
IN SPREADSHEET FORMAT**



The database created for TxDOT was designed to be very user friendly and easily updatable. When the program is started the user is greeted by the screen shown in Figure D.1. At this point the user can:

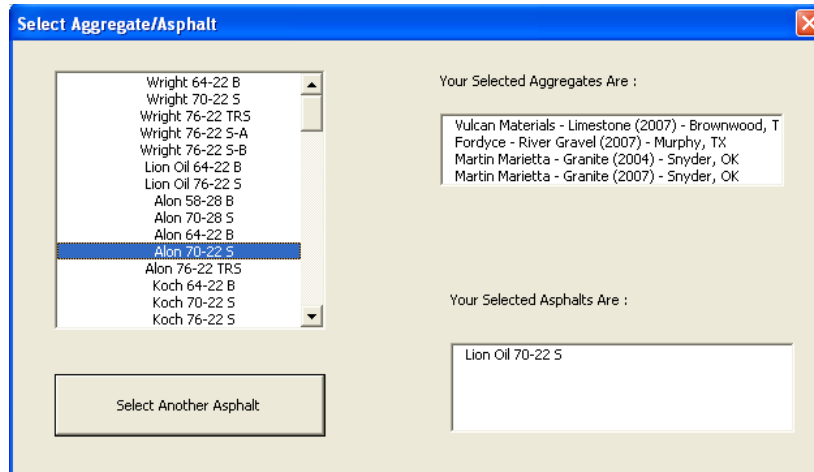
- Analyze existing materials in the database;
- Add new asphalt or aggregate information into the database;
- Delete an existing asphalt or aggregate out of the database; and
- Exit the user interface and navigate the spreadsheet.



**Figure D.1. User Interface.**

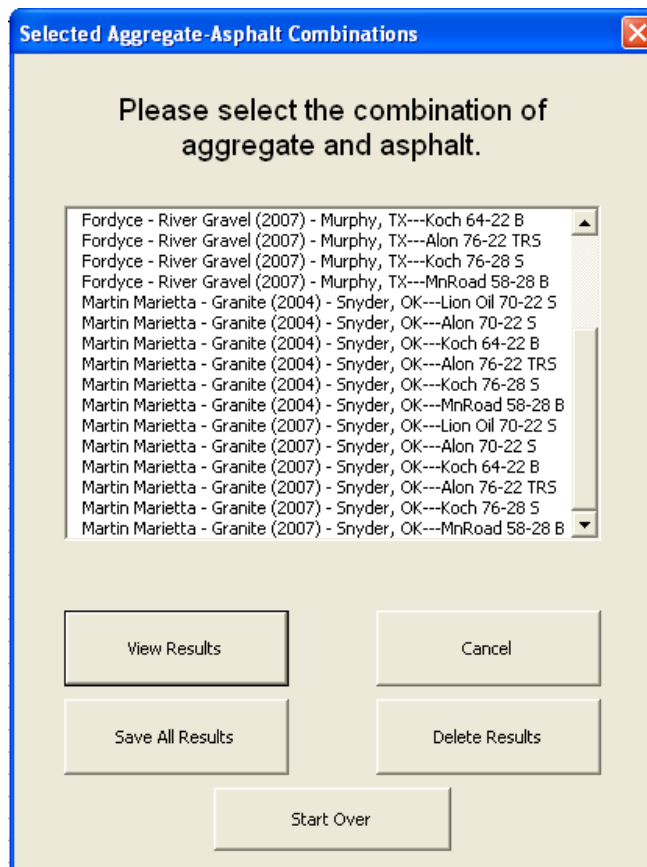
### **Analyze Available Aggregate-Asphalt Combinations**

This option will allow the user to select as many combinations of asphalt-aggregate as are contained in the database. Figure D.2. below illustrates the selection screen. All asphalts and aggregates are displayed in this screen and disappear once they have been selected.



**Figure D.2. Asphalt-Aggregate Selection Screen.**

When the user has selected the asphalts/aggregates they are interested in, a screen showing all possible combinations is displayed, as shown in Figure D.3.



**Figure D.3. Combinations of Asphalt-Aggregate Chosen by User.**



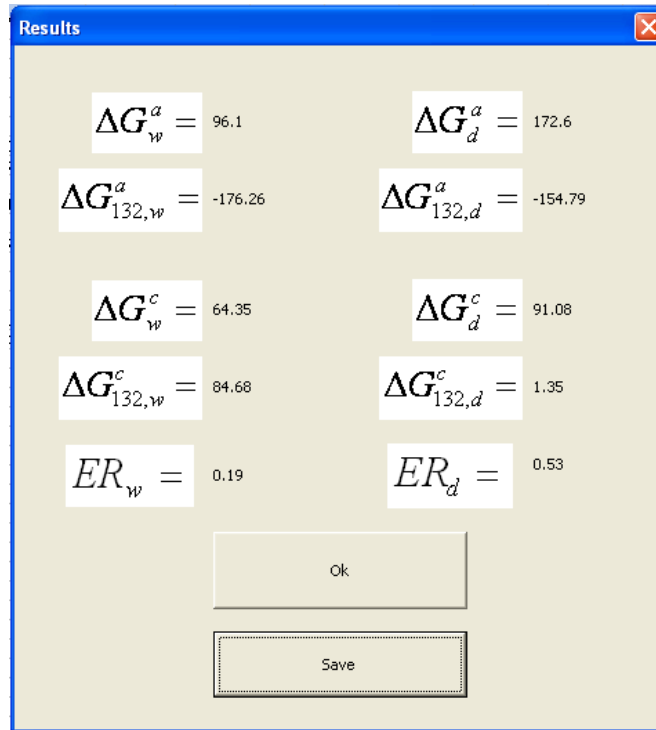
The user now has the options of:

- Viewing the results of any asphalt-aggregate combination;
- Saving the results of all combinations in the “Results” worksheet;
- Exiting the user interface mode;
- Deleting results from the “Results” worksheet; and
- Starting back over from the beginning screen shown in Figure D.3.

If the “View Results” button is clicked, a screen showing the results for the selected asphalt-aggregate combination is displayed; an example is shown in Figure D.4. These individual results can be saved in the “Results” worksheet, or the user can return to the previous screen.

Clicking the “Save All Results” or “Delete Results” will either save the current information into the “Results” worksheet or allow the user to delete any or all the information out of the “Results” worksheet, respectively.

The “Results” worksheet, shown in Figure D.4. below, organizes the results seen in Figure D.5. in a tabular form. The user also has the option of printing the results, saving the results in a separate workbook without the program, or restarting the user interface.



**Figure D.4. Results from Asphalt-Aggregate Combination.**

S.No	Combinations	$\Delta G_w^a$	$\Delta G_d^a$	$\Delta G_{132,w}^a$	$\Delta G_{132,d}^a$	$\Delta G_w^c$	$\Delta G_d^c$	$\Delta G_{132,w}^c$	$\Delta G_{132,d}^c$	$ER_w$	$ER_d$
1.00	Fordyce - River Gravel (2003) - Murphy, TX---Alon 64-22 B	83.03	155.22	-322.79	-305.63	64.35	91.08	84.68	1.35	0.06	0.21
2.00	Texas Industries - Limestone (2004) - Bridgeport, TX---Alon 64-22 B	96.10	172.60	-176.26	-154.79	64.35	91.08	84.68	1.35	0.19	0.53

**Figure D.5. Results Worksheet.**

### Add a New Asphalt/Aggregate

When adding a new asphalt or aggregate the program will ask you to supply specific information about the material being added. With this information, the program can keep itself more organized and prevent duplicate information from being accidentally added. It is recommended that all information added is done through the user interface. Once an asphalt or aggregate has been added, the program will automatically save to

prevent information from being lost. Figures D.6. and D.7. below illustrate the templates for adding asphalt and aggregates, respectively. When adding an asphalt, the program will ask for both the wetting and dewetting values. The wetting values refer to the surface energy components computed from contact angles generated when the asphalt was being immersed into the probe liquid. The dewetting values refer to the surface energy components computed from contact angles generated when the asphalt was removed from the probe liquid.

The figure shows two side-by-side dialog boxes. The left dialog, titled 'Asphalt Name', prompts the user to enter details for an asphalt. It contains four sections: 'Asphalt Name' with a text input field; 'Performance Grade' with two input fields separated by a hyphen; 'Modifier' with radio buttons for Base, SBS, SBR, Tire Rubber, and Other, plus an empty text input field; and 'Aging' with radio buttons for Unaged, PAV, SAFT, RTFO, and Other, plus an empty text input field. The right dialog, titled 'Gamma Values', prompts for different gamma values. It has two columns: 'Wetting' and 'Dewetting'. Each column has three rows for 'Non-Polar (Gamma LW)', 'Acid (Gamma +)', and 'Base (Gamma -)', each with a corresponding text input field. Both dialogs have 'Ok' and 'Reset' buttons at the bottom.

**Figure D.6. Asphalt Input Sheets.**

The figure shows a single dialog box titled 'Aggregate Details' with the prompt 'Please Enter the details of the Aggregate to be added.' It contains four main sections: 'Aggregate Name' with a text input field; 'Location' with a text input field; 'Non-Polar (Gamma LW)' with a text input field; and 'SSA (Optional)' with a text input field. Below these are three more rows for 'Acid (Gamma +)' and 'Base (Gamma -)', each with a text input field. At the bottom are 'Ok' and 'Reset' buttons.

**Figure D.7. Aggregate Input Sheet.**

**Delete an Available Asphalt/Aggregate**

Any asphalt or aggregate may be deleted by using the user interface. This should be done with caution because once the asphalt or aggregate has been deleted, the program will automatically resave, making any deletions permanent.

**Cancel**

Pressing the cancel button will exit the user interface mode and allow free access to the spreadsheet. Adding additional asphalts or aggregates outside of the user interface is strongly discouraged. Duplicates could be added or values misplaced, which could compromise the functionality of the database. In the “Results” worksheet the user is encouraged to view the results, save data, or print the results for his/her records.