

A SIMPLIFIED METHOD OF
MEASURING THE SURFACE TENSION
OF ASPHALTIC OILS AND
COMPARATIVE TESTS OF THEIR
BONDING PROPERTIES

Thesis for the Degree of B. S. William H. Smith 1937

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# A SIMPLIFIED METHOD OF MEASURING THE SURFACE TENSION OF ASPHALTIC OILS AND COMPARATIVE TESTS OF THEIR BONDING PROPERTIES

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## TOREWORD

In propering this thesis the author wishes to seknowledge the helpful criticisms and hints that were given by Professor E.A. Finney of the Civil Engineering Department of Michigan State College, and T. Wolenyaski, the laboratory assistant of Michigan State Highway Department, also to acknowledge indebtedness to the Civil Engineering Department and the Michigan State Highway Department for the use of the apparatus and materials, without which, this work sould not have been completed.

### INTRODUCTION

Within recent years the application of asphaltic cile to the improvement and construction of roads and highways has become one of the forement industries of this country. Although up to the present time there has been very little direct research carried on with the sole purpose of determining the properties or conditions which help these cile become more efficient in their application to the roads, it is my belief that in order to have an efficient road cil certain important bilineaive properties must be present. Since there are numerous methods of changing these properties, it is possible that cile may be comparatively improved by a simple change in any one or more of these properties. However, it will not be the purpose of this thosis to find out the changes that may be made, but its purpose is to determine some of those properties and simple methods of measuring them. The proper changes and methods are left for later research work.

In their research reports, A.R. Lee (1) and C. Mack (2) stress the important energy relations between asphalts and aggregates. They state that these relations, energy of adhesion, interfacial energy, and spreading coefficients of the cils with aggregates, was, are with slight variations, in proportion to the surface tension of the asphalts and aggregates. Assuming that these relations are true, it is the purpose of this thesis to device a simple means of determining the surface tension of asphalts and to test the asphalts to see if certain properties shown by the energy relations may not scandar

predict the conditions that will be present when the cils are used in a mix with the tested aggregates.

Throughout this thesis, the words asphaltic oils, asphalts, and oils, will be used interchangeably to mean the same thing.

Since the surface tension of liquids is very important in this thesis. I believe a definition of Surface Tension is well in order.

On the Molecular Theory of Matter, each molecule within a liquid attracts and is attracted by surrounding molecules. Each molecule may thus be pictured as surrounded by a sphere of attraction. Forces them exist between each molecule and other molecules within its subere of attraction. In the body of a liquid the resultant of these ferces is more, since each molecule is completely surrounded by similar molecules: in the surface layer this does not hold, as the molecules in the upper half of the sphere of attraction are comparatively few. Conscioutly, the free surface of a liquid behaves as if it were in a state of stress. Forces act agrees the surface which are the direct result of melocular attraction and consequently their measurement is of importance. If a straight lime I on, in length is drawn in the surface of the liquid in contact only with its own vapor, then the force agrees this line at right engles is defined as the Surface Tension of the liquid. Surface Tension is thus force per unit length. - (Physico-Chemical Methods - Reilly, Pg. 886)

Up to the present due to their various degrees of tenacity and matural color, there has not been devised a simple means of determining the marface tenaion of asphaltic oils. Of the various ways which the surface tenaion has been determined the one most applicable to general practice is given by C. Mack, (2)

He suspends a thread of asphalt having a known radius and length under conditions where only the forces of gravity and surface tension may not on it. After a certain length of time the action of gravity and surface tension are rated and through compilation of certain data on several strings of asphalt, he is able to determine the surface tension of the oil. There is only one serious objection to this method, namely, it can be applied only to asphalts which flow under the influence of gravitational ferces. One of the semewhat less serious objections is the necessary time consuming operation of accurately measuring the radius of the thread.

The simple method I propose to be used hereafter is based on DuNouys Ring method of obtaining surface tensions of liquids. Although the ring method has been previously used to determine the surface tension of oils at high temperatures by Nellensteyn and Roodenburg, this method is highly influenced by the viscosity of the oil. Hence, it may give inaccurate results.

Since all asphalts are soluble in Benzene, and the surface tension of Benzene is easily measured with Du Houy's ring, it is a simple matter to mix different weight proportions of Benzene and oil, thus determining the surface tension of these mixtures with the ring. Since all the solutions with percentages ranging from 45-89% Benzene are extremely liquid, one has no trouble measuring the surface tensions.

the precedure is very simple and routine. A small sample of the cil (5-5 grs) is weighed in an Erimeyer flask or other bottle which has been weighed with a cork to fit. To the flask is added an approximate essent of Benzene which will comprise from 45-90% of the solution, the flask is corked and accurately weighed again to determine actual percent of Benzene. Three or more samples are weighed out and their Benzene percentages is spread evenly as possible between 45 and 95%. (Note. For percentages less than 45% Benzene the solutions are selden sufficiently liquid, and slight difficulty may be encountered then eleming the platinum ring, also an infinitesimally small error may occur due to the cil actually stitking to the ring, in addition percentages above

With the accurate percentages of Bennene in each solution determined, the flasks are carefully rotated to guarantee a uniform mixture. Then a small watch glass, intensively closed and rinsed beforehand is filled und the surface tension of this sample is obtained with an accurately caliberated DaNouy Tensioneter. Since three or more samples are taken from each solution, the flasks are kept corked between trials. The operation of the Tensioneter is neither intricate nor complex, if used as per instructions given with the instrument. The ring is closed after each trial by heating red hot in a yellow flame. The mean Surface tension of the three trials is taken as the actual value of surface tension for each solution. These values are taken as ordinate values and plotted with the Bennene Percentages as abscisses, the curve will actually be a straight line. With the least error possible the curve will pass through the points of the surface tension of Bennene at a corresponding value of 100% Benzene. By extending the

sealed will be the value of the surface tension of the asphalt.

The results for oil Bard shows - Table

# Beamens 100.0 88.7 79.5 72.6 50.8 0
Surface Tension 28.87 80.01 20.07 20.51 50.15 51.80

## Mergy Relations

Asphalt through its property to act as a binder for aggregates has found its greatest field of application in road construction and improvement.

In an asphalt-aggregate system, the surface energies play an important part. These energies are given certain differentiating names and shell hereafter be called, Interfacial Energy, Energy of Immercian, Energy of Adhesion, Energy of Cohesion and Spreading Coefficient. Definitions of these terms are taken from Mack (1) and Amatole (4).

Interfacial Energy The energy at the interface of two coalescent substances. By Andrew rule it is the difference between the surface tensions of the unterials. R. Lousa (5) and N.P. Zwikker (5) show that this rule holds best for the solid-liquid interfaces.

Energy of Immersion When a solid is immersed in a liquid the surface

disappears and in its place is found the same area of solid-liquid interface. The energy of immersion is equal to the surface tension of the immersed particle minus the energy of the interface.

82 \* Surface Tension of Aggregate

KI = Interfacial Energy

Energy of Immersion = SZ - EI (Mack)

44.4

in a second of the second of t

Mercy of Adhesion Whenever a solid coalesces with the mrfaces of a liquid the surface energies disappear and the energy of the interface appear. The energies involved in this reaction is equal to the sum of the surface tension, ninus the interfacial energy.

Energy of Adhesion = 8Z +80 - EI

The energy of Adhesion is the emount of energy per on which must be applied at the interface in order to separate the substances.

Cohesion Energy is the internal energy between molecules of the some substance. This attraction energy is assumed to be twice the surface tension.

So = Surface Tension of Oil

Energy of Cohomica = 8 So.

The Spreading Coefficient. When a liquid spreads ever a solid, attraction between the two phases occurs, the force of which is directed against the chhecion of the liquid, which is in a position of spreading. Thus the coefficient of spreading is equal to the energy of adhesion minus the energy of echesion.

Coefficient of Spreading = SZ - SO - KI

By solving Mack's equations for adhesion tensions, the mrface tensions of limestone is found to be approximately 71 dynes. Since this experiment is solely to compare the adhesive energies of the cile, and any assumed error is constant for all cases, the suthor believes that a const value of 70 dynes / on is a suitable value for the surface tension of limestone. The following table shows the comparative calculated results.

	Surface	Energy of	Energy of	Energy	of Energy of	Coefficient of
OIL	Tension	Inteface	Immersion	Adhesio	n Cohesion	Spreading
A	37.2	-32,8	102,8	140	74.4	65.6
B	31.8	-38.2	108.2	140	63.6	76.4
C	31.9	-38.1	108.1	140	63.8	76.2
D	31.9	-38.1	108.1	140	63.8	76.2
E	33.2	-36.8	106.8	140	66.4	73.6
F	31.6	-38.4	108.4	140	63,2	76.8
G	35.3	-34.7	104.7	140	70,6	69.4
H	33.8	-36.2	106.2	140	67.6	72.4
I	32.1	-37.9	107.9	140	64.2	75.8
J	32.9	-37.1	107.1	140	63.8	74.2
K	32.3	-37.7	107.7	140	64.6	75.4
L	32.8	-37.2	107.2	140	65.6	74.4
M	32.5	-37.5	107.5	140	65.0	75.0
N	32.3	-37.7	107.7	140	64.6	75.4
•	33.6	-36.4	106.4	140	67.2	72.8
P	31.8	-38.2	108.2	140	63.6	76.4

Remarks: Since it is practically impossible for all the oils to have the same adhesion energy, it is obvious that at least one or both of previous assumptions; namely, the first assumption, Antendada Rule must hold for asphaltic oils and aggregates; second, that the surface tension for limestone can be assumed to have a constant value of 70 dymes: ere false.

No general conclusions can be made from the data of the preceding paragraphs for the reasons previously stated. The author regrets that he cannot find any appreciable error in his computations; and, until further research is carried out along this line, he will be unable to state definitely which of his assumptions is incorrect.

## Binding Properties of Asphalts

The Attribute of an adhesive is that it should wet the solid on which it is placed. It should achange into a more or less tenacious solid. In road construction the asphalt-aggregate joint is

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usually attached by water before setting of the adhesive occurs. The first stages in the study of binders should be in the wetting properties. Since asphalts spread easily over most aggregates, wetting is comparatively simple and complete. But, nevertheless, the degree of wetting depends on (a) the nature of the liquid which will determine the specific wetting properties, (b) the viscosity of the liquid which will determine its rate of spreading, (c) the mature of the solid which will determine its tendency to be spread.

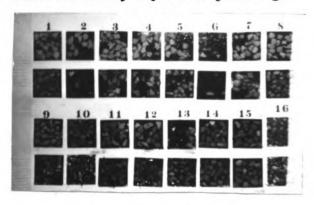
The second stage of study of binding properties is their condition after being water-scaked for some definite length of time.

A Glass Plate test is used to investigate the binding properties of asphalts after soaking. This test is accomplished by coating two pieces of glass plate (in this instance, three inches square) with oil films of uniform thickness. Granite and limestone. "in size, which had been previously soaked for 24 hours and wiped dry with a clean coth, was placed on the two plates. One plate for each specific aggregate. Samples of sixteen different oils were spread on similar plates. After allowing the glass plates to remain in the open for fifteen minutes, they were placed in a container which had enough water to cover them to the depth of about three inches. This water was kept at room temperature (20°C) throughout the test. These plates were allowed to soak for ten days, after which the glass plates were quickly inverted and held in this position to allow the stones whose bonds had been broken by the water to fall off. Through this test a comparative binding efficiency of the oils for granite and limestone is made. Also, under normal

from the plate glass surface. With each granite plate having almost the same film thickness as the corresponding limestone plate, the results were, as expected, very much alike.

Taken as a whole, the majority of the oils showed exceptionally good Bond for the limestone, with a few showing likewise for granite. The exceptionally poor oils were: F, approximately 2% efficient as a bond agent or, it was almost totally displaced from the stones by the water; oil M was an exception in the way it acted as suitable binder for the grainite plate, but not for the limestone. This case was opposite to that of oil B, which showed good Bond for the limestone. Another characteristic of almost all of the plates was the condition of the oil film on the glass plate: with a few exceptions, the films were all broken and formed into weathered, disentegrated oil masses.

The exceptions spoken of in the previous paragraph were oils G, H, I, K, L, N, and O. They showed the least film weathering and practically no displacement of oils from either the stones or the glass plates. Taken as a comparative test, the plates showed the best oils for cold, dry stone patching.



The preceding snapshot shows, although none too clearly, the black plates—the ones whose oil bond was insufficient to hold the stones. Reading left to right, the top row beings with the lime—stone plate of oil A and so on to oil H. The plates in the second row are the corresponding granite plates of the oils. The third row begins with oil I on the left and ends with oil P on the right, with the corresponding granite plates below them. The following table compares the binding properties of each oil.

PLATE TEST - TEN-DAY SOAKING

011s	Limestone Bond	Granite Bond	Film Weathered
A	Good	Good	Slightly
В	Poor	Very poor	Highly
C	Good	Good	Slightly
D	Very good	Good	Slightly
E	Fair	Fair	Highly
r	Exceptionally poor	Except. poor	Highly
G	Very good	Very good	Very slightly
H	Perfect	Perfect	Not weathered
I	Very good	Very good	No weathering
J	Good	Very good	No weathering
K	Poor	Very good	Slightly
L	Very good	Perfect	Slightly
M	Very poor	Very good	Slightly
N	Very good	Perfect	No weathering
0	Perfect	Perfect	No weathering
P	Very good	Fair	Highly -
			•

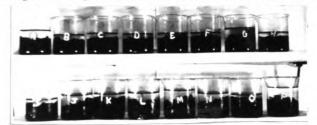
Remarks: The various grades of bond for each oil is obtained by comparing the number of stones that remain on the plate after soaking, with the number originally placed on it before soaking. The weathered condition of the film denotes the degree of destruction of the film by the water.

In order to study more clearly the effects of displacing and weathering of the oils by water, a Beaker Test was devised to com-

plete the experiment. It consisted of samples of 150 gms of "1" limestone being coated with 1-1.5 gms of oil. In order to increase uniformity of films, these samples were warmed to 100°C, allowed to cool for thirty minutes, then placed in numbered beakers and covered with distilled water. These beakers were set in a place well out of the sunlight and allowed to soak. They were inspected of ten enough to be sure that the stones were completely covered with water at all times.

After fifteen days of soaking, the beakers were inspected. With few exceptions, the oils had formed spherical drops on the stones.

Oils B, G, and M, in addition, showed a slight change in color from black to slightly brown. Although the results were easily noted on sight, it was quite impossible to show distinct effects by snapshots.



On close scrutiny, the stones in beaker F appear much lighter than the others. This was caused by the almost complete displacement of the oil from the stones by the water. The following table shows the comparative results after a thirty-day period of soaking.

BEAKER TEST - THIRTY-DAY SOAKING

Oils	Color	1	Condition of Film	1	Luster
A	Dark brown		Fair		Glossy
В	Light brown		Fair		Dull
C	Brown		Poor		Slight gloss
D	Black		Good		High gloss
E	Dark boown		Fair		Glossy
F	Dark brown		Very poor		Slight gloss
G	Light brown		Good		Dull

# BEAKER TEST - THIRTY-DAY SOAKING (con't)

H	Black	Very good	Clossy
1	Brown	Good	Dull
7	Brown	Very good	Dull
K	Black	Fair	High gloss
L	Black	Good	High gloss
X	Very light brown	Fair	Dul1
M	Light brown	Very good	Dull
0	Black and brown	Very good	Partly glossy
P	Light brown	Fair	Du11

Remarks: Only those oils whose color remained black were considered to be non-weathered. Also the films of these oils were in either very good, or at least fair, condition. The film conditions were graded as to the completeness of the present oil film on the stones. The oils having very poor or only fair conditions show oil displacement by water.

## Acid Test

In order to complete the analysis of the oils and to test the influence of acid on their properties, they were tested for their acid (7) content.

A small sample (5 gms) of the asphalt was boiled with a retort in 25 c.c. of benzene. Then 100 c.c. of slightly meutral alcohol was added to the retort and boiled for twenty minutes longer. The clear solution was decanted and 50 c.c. more alcohol was added to the residue, then boiled twenty minutes more under setort. This solution was decanted and the residue tested to make sure it was no longer acid. Then the total clear solution was mixed and 10 c.c. of barium chloride was added. After allowing it to cool, six drops of phenolphthalein were added to the solution as indicator, and it was titrated with .64% mormal NAOH.

The Acid Equivalent was found to be equal to the Mgs NAOH necessary to neutralize 100 gms of oil.

Example: 011 B

Sample 5.0932 gms. - used .1 cc. .002593 gNAOH/cc

1x.002593 x 100 x 1000 = 5.096 mg/100 gms oil 5.9032

TABLE I

	Character	istics o	f Oils			
PROCESS	Steam Refi Residue	ned Cut	Back am Resi Gas 01	due fine	d of St d and C Residue	eam Re-
OILS	J	F	C	L	В	E
Specific Gravity	Married Street, Street				ended constitution	
25°C/25°C	.972	.983	.97	8 1,025	.992	.995
Water & Sediment %	0.0	0.0	0.4	0.0	0.3	0.0
Flash Point-Cleveland						
Open Cup OC	156	150	14	8 154	130	145
Viscosity-Saybolt Fur		7				
60°C Sec.	522	552	46	5 472	446	649
Heterogeneity Test	Negative	Negat	ive Ne	g. Posit	ive Sli	Pos Sli P
Distillation °C						
00 - 2250 0/0	0.0	0.0	0.0	0.0	0.0	0.0
00 - 3150 0/0	0.0	0.5	0.2	Trace	1.5	0.0
00 - 3600 0/0	1.5	3.0	7.0	1.0	7.0	0.5
Test on Distillation Residue						
Bitumen Soluble in						
0Cl_ °/o	99.94	99.98	99.99	99,89	99.93	99.97
Tests on 100 Penetrat Residue	ion.					
Actual Penetration of						
Residue	95	107	93	100	105	93
Heat Time for Residue						1112
Hrs.	7	92	6	3	11	112
Total Residue º/o	79.7	82.3	78.8	80.1	79.1	80.1
Ductility 25°c 5cm/mi	n.					
cmo.	95	190	111	148	100	110
" 4°C "1 "	7	9.5	6.5	7	5.5	6.5
Asphaltenes %	10.	8.61	12.66	19.66	8.95	
Surface Tension Dynes	32.85	31,56	31.88		31.81	
Plate Test -Limestone	Good	V.Poor	Good		Poor	Fair
-Granite	V.Good	V.Poor	Good	Perfect	V.Poor	Fair
Bealer Test-Limestone	V.Good	V.Poor	Poor	Good	Fair	Fair
Acid Equivalent - MG/100	14.23	43.54	25.43	4.87	5.10	23.92

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# TABLE I - Continued

# CHARACTERISTICS OF OILS

Process	Steam	and Vac	na Distil	la tica	<del></del>	
011	! D	G	1	K	. 1	
Specific Gravity 250/25	C .989	.984	.990	1,000	556	.976
Water & Soliment %	0.05	6.05	0.05	0.08	0.05	0.00
Flash Point Cleveland						
Open Oup O	<b>834</b>	140	340	180	174	206
Viscosity-Saybolt Farol						
60°C Sec.	480	<b>595</b>	496	435	685	552
Heterogenel ty Test	Nog.	Nog.	Nog.	Nog.	Nog.	Nog.
Distillation of						
0° - 225° \$	0.0	0.0	0.0	0.0	0.0	0.0
00 - 8150 %	0.0	8.0	Traco	Trace	0.0	0.0
00 - 5600 %	0.5	12.0	2.5	1.2	Trace	0.5
Test on Distillation						
Residue						
Bitumen Soluble in						
00/4 \$	99.94	99.91	99.95	99,95	99,95	
Tests on 100 Penetration	B					
Residue						
Actual Penetration of	00	100	00	-		
Residue	90.	107	99	96	98	
Heat Time for Residue Hrs.	22	8	•	4.78	•	
Total Residue \$	75.5	75.5	76.2	77.4	76.3	
Dustility 2000 Sen	70.0	70.0	70.5	77.4	70,0	
1 min. ems.	148	126	106	100	94	
" 4°C Sem 1 min.	400		200	. 200	•	
	7.0	9.5	9.5	7.0	9.0	
Asphaltenes \$	6,46	20.41	15.14	11.78	14.07	5.08
Surface Tension Dymes/em.	21.02	85.26	58.11	82,55	52,45	<b>51.75</b>
Flate Test - Limestone	V.Good	V.Good	V. Good	Poor	Y.Poor	Y.Good
- Granite	Good	V.Good	V.Good	T.Good	-	Pair
Booker Tost - Limestone	Good	Good	Good	Tair	Tair	Tair
Acid Equivalent	51.35	4.66	14.27	14.21	9.83	19,58

TABLE I
CHARACTERISTICS OF OILS

Process	Blended Cracked R	esi dues	Straie Crack		
Oile	X	H	<b>A</b> :	0	
Specific Gravity 25°c/25°C	.999	1.053	1.082	1.051	
Water and Sediment %	0.00	0.02	0.00	0.40	
Flash Point Cleveland Open		3432	3,00	0000	
Cam oC	170	155	152	210	
Viscosity Saybolt Furol					
60°C, See.	637	467	480	456	
Heterogeneity Test	Positive	Positive	Positive	Positive	
Distillation					
0° to 825°C %	0.0	0.0	0.0	0.0	
0° to 515°C "	Trace	Trees	Trace	Trees	
00 to 360°C "	0.5	8.5	1.8	0.5	
Test on distillation Residue					
Bitumen Soluble in CCl.	99.94	99.85	99.96	99.52	
Tests on 100 Penetration	0000		00000		
Residue					
Actual Penetration of Residue	91.	94	96	107	
Heat Time for Residue Hrs.	4.5	2.5	1.5	7	
Total Residue %	81.8	61.8	94.0	84.6	
Duetility 25°C Sem 1 min.Cms.	150	158	106	70	
# 4°C # 1 #	5.5	1.0	0.0	4.5	
Asphaltenes - Percent	13.04	19.05	26.00	15.80	
Surface Tension - Dymes/cm.	52.50	83.77	57.19	85.55	
Plate Test Limestone	V. Good	Perfect	Good	Perfect	
Granite	Perfect	Perfect	BooD	Perfect	
Beaker Test - Limestone	V. Good	Y. Good	Tair	Y. Good	
Acid Equivalent	10.55	4.91	9.41	26.92	

## SUMMARY AND CONCLUSION

- The DuNouys Ring and Percent Benzene method, if accepted, is the most practical yet proposed for determining surface tensions.
- 2. Antonows Rule, although valid for pure liquids, does not hold for asphalts and aggregates.
- 5. The Plate Test is a simple, but effective, test for the bonding properties of asphalts.
- 4. The Beaker Test is by far the most efficient of the tests on oil displacement by water.
- 5. The oils having the highest surface tension were either crecks or blends of cracks. The only exception to this conclusion is oil G, which is a steem refined crude.
- 6. Oils having low surface tensions were found to have fairly poor bonding qualities. However, this is not true for oil D.
- 7. Oil F is an exceptional example, having the lowest surface tension. It is also outstanding as the poorest binder of the oils.
- 8. Compared according to their asphaltene content, the oils with the highest surface tension also have the greater asphaltene content.

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