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UNIVERSITY OF LOUISVILLE

A STUDY OF THE COHESIVE AND ADHESIVE PROPERTIES OF ASPHALT

A Thesis

**Submitted to the Faculty
of the Graduate School
of the University of Louisville
in Partial Fulfillment
of the Requirements
for the Degree of**

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

James
Harold Rose

1940

A STUDY OF THE COHESIVE AND ADHESIVE PROPERTIES OF ASPHALT

HAROLD ROSE

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V

ABSTRACT

A study was made of the adhesive and cohesive strengths of various blown asphalts and an attempt was made to correlate these values with the commonly accepted physical tests, softening point, penetration, and ductility.

A method of measuring the adhesive and cohesive strengths of asphalts was developed and is described.

Curves of adhesive and cohesive strength against temperature are plotted. These curves give an indication of the adaptability of the asphalt for cementing purposes. A method of this type could be used in writing specifications of asphalts for uses as an adhesive.

INTRODUCTION

A measure of adhesion or cohesion, together with other accepted physical determinations, is of value in determining the adaptability of an asphalt for certain definite purposes, such as for paving, road building, the manufacture of compounds for waterproofing and built-up roofing work, and cements. Each industry usually has its own particular test for these two properties depending upon the type of work in which the asphalt is used. It would be advantageous to correlate cohesion and adhesion with other accepted empirical tests on asphalts, such as penetration and softening point.

The purpose of this investigation is to study the adhesive and cohesive strengths of various air blown petroleum asphalts.

It was proposed to study these two properties by (1) obtaining curves for the variation of adhesive and cohesive strengths of asphalts manufactured from different crudes with temperature and (2) reviewing the results obtained from these curves with respect to some of the existing empirical properties, such as penetration, softening point, and ductility.

A thorough search of the literature revealed that very little work has been done on this subject.

HISTORICAL

The term "asphalt" may be traced back to Babylonian times. It was later adopted by the Homic Greek in the form of an adjective meaning "firm, stable, secure" and in a verb form meaning "to make firm or stable, to secure." It is known that the first use of asphalt by the ancients was in the form of a cement for holding together various objects, and thus it seems that the name asphalt was expressive of its use (1).

The earliest recorded use of asphalt by the human race was in the nature of an adhesive by the Babylonians in the Euphrates Valley (about 3000-2500 B. C.). These people, skilled in carving and decorating stone used asphalt to cement bits of stone and shells in place (19). About the same time, the early Persians were also using asphalt as a material to cement eyes in alabaster figures (7).

The floor of Nebuchadnezzar's Temple, Babylon, was made of brick held together by means of asphalt (16) and are still so firmly joined together that it is impossible to separate the bricks without destroying them.

From these times forward records diminished except for mentioning several sources of asphalt. The discovery of the asphalt lake in Trinidad in 1595 by Sir Walter Raleigh (18) started the modern history of bitumens. M. Secretan in 1797 obtained a concession from the French government to work the asphalt deposits at Seyssel on the Rhone, France. In 1802, Count de Sassenay, actively exploited the deposits and a laboratory was built to investigate the uses of this asphalt.

In 1835, the first asphalt master pavements composed of Seyssel asphalt were laid in Port Royal, Paris (10). In 1836, London followed the example set by France the year before. In 1838, asphalt was first used in the United States for foot pavements in Philadelphia, Pennsylvania. Seyssel asphalt was used for this purpose.

The first compressed asphalt roadway, a short stretch of road, was laid in Paris by M. Vaudrey (6) 1854. In 1858, a long stretch of asphalt roadway was constructed on the Palais Royal and on the Rue St. Honore in Paris. The first compressed asphalt pavement in London was laid at Threadneedle Street near Finch Lane in 1869. It was composed of Val de Travers rock asphalt. E. J. Smedt, who is credited with having built the first rock asphalt roadway in the United States, laid a small strip of asphalt opposite the City Hall in Newark, New Jersey. This rock asphalt was said to be too slippery, and in 1876 the first Trinidad asphalt pavement was laid in the United States on Pennsylvania Avenue, Washington, D. C. (4). This asphalt is still widely used today in road construction.

Although asphalt has been used for several thousand years in various manners, only a very few scientific investigations were made on this material until the last part of the 19th century. From 1900 to the present a great deal of work has been done on the composition, origin, and physical and chemical properties. Since about 1915 the emphasis of scientific investigation has been directed towards industrial uses as road building, mastics, emulsions, insulating compounds, and protective coating.

Asphalt is a series of very complex organic compounds and does not readily yield to absolute methods of analysis or control. As a consequence, therefore, the major portion of current physical tests are empirical, and of those test apparently chemical in nature, the majority are interpreted empirically.

At least two of these tests have general research and industrial acceptance, namely, penetration and softening point. Even here there is some disagreement concerning the proper method of carrying out these results. But mainly through the impetus of the American Society of Testing Materials and American Association of Highway Officials these tests are largely standardized. The indications given by these two tests, however, are not necessarily satisfactory for specific problems but serve mainly as a method of classification.

Spielman (21) says

"It is of interest and importance, both for scientific and practical purposes, to determine whether and how far one property varies regularly with another or is entirely independent of it. This subject requires more attention to be paid to it than has already been given.

"In some cases clear correlation is apparent, sometimes this is doubtful, but more frequently there is no traceable connection between the figures obtained."

Spielman compared ten of the most used properties (test characteristics) and obtained the following results:

	Per cent
Obvious correlation	42
Doubtful correlation	7
No correlation	51

Lewis and Welborn (13) attempted a correlation of a majority of the current physical and chemical properties of various asphalts and

concluded that

"It would seem from a consideration of the known service performance of similar materials and from a study of the test data presented in this report that these special tests are essentially tests that assist in the identification of source or the method of processing or that they are measures of special characteristics. There has been no definite correlation between the test data on asphalts and their performance in service to indicate that any of the tests discussed in this report are true measures of quality or durability. Specifications for asphalt cement, therefore, will remain inadequate until such correlation is provided or more satisfactory tests are developed."

The latter report (15) is recommended as a compendium of current tests.

All asphalts have the property of adhesion to a varying degree. For this reason, they have wide industrial use, as in the building of roads, manufacture of waterproofed paper, foil coated papers, mastics, inexpensive molding plastics, and water-proofing membranes.

Because of the importance of asphalt in road construction many tests have been devised to indicate probable performance in which the adhesion is considered as an important function.

The Riedel and Weber (20) adhesion test in which asphalt coated stone is boiled in soda solutions has been modified by Holmes (11) who replaces the boiling soda solution with boiling distilled water; after one minute immersion the sample is cooled under running water and spread out. The amount of coated surface is noted and is claimed to be a measure of adhesion.

Russel and Neumann (17) tested the adhesiveness of bitumen-coated stone by using the Riedel and Weber (20) test and the water displacement test (11). Because the two methods did not agree they stressed the importance of the surface properties of the filler.

A water displacement test by Holmes (11) in which asphalt covered aggregate is immersed for 18 to 20 hours in distilled water at 140° Fahrenheit, and the adhesion measured as in preceding test, agreed fairly well with the water boiling test.

Holmes (11) developed a circular track test for asphaltic road material in which the asphalt was subjected to accelerated weathering and as near actual road conditions as possible. By correlating the water displacement test on asphalt with its circular track test he found that road life was increased with increased asphalt adhesion.

Hoover and Shafer (12) in measuring the adhesion of asphalt coating, directed a jet of water from a nozzle to impinge at the edge of an isolated area of the coating material. The coating support and nozzle were at an angle of about 45°.

Oscar G. Goldman (9) in 1935 patented a method of testing the bond of an adhesive protective coating in situ as on large steel pipes, plates, and shapes. The method consisted in stripping the coating from a surface at an angle of 90° by means of a machine in which the force applied was read on a gauge.

The direct measurement of adhesion of asphalt is not yet soundly established. A method (8) has been described in which an asphalt sample is "smearred on" two brass discs one inch in diameter and pressed firmly together. One is held immovable while the other is fixed to a cord which passes over a pulley and is attached to a small pail. Into this pail shot are poured regularly at a rate of 2,250 grams per minute and the weight noted when the plates are pulled apart at a temper-

ature of 20° Centigrade. This method would appear to be subject to the difficulties of controlling temperature and avoiding shear and torsion effects on the sample.

Osborne developed a test for adhesion (1) in which a uniform layer of the material to be tested is placed between two concentric cylinders, whose temperature is controlled by circulating water through the inner cylinder. The thin layer of bituminous material between the cylinders offers resistance to turning. The test seems, however, to measure viscosity rather than adhesion.

Another method (2) is to melt the sample of asphalt into a split cylinder and pull the halves apart in a dynamometer. The force necessary to pull the two halves apart is a measure of the adhesion.

Cohesion of asphalts is determined solely by means of its tensile strength (1). This is recorded on a tensometer (1) and is equal to the maximum reading in kilograms as the two halves of the "hour-glass" mold separate.

THEORETICAL

McBain and Lee (15) define an adhesive as "any liquid material which wets adjacent surfaces and which could be solidified 'in situ'."

The theory of adhesives depends in part upon the hardening of the cementing material and its adhesion to a surface (3). One adhesive may be used for a number of different materials while others give good results only for special materials.

McBain and Hopkins (14) distinguish between two kinds of adhesive joints, mechanical and specific. Mechanical joints are those in which the agglutinate is mechanically embedded in the pores at the surface of a material. The strength in this case depends largely on the cohesion and not on adhesion alone. Specific or true adhesion occurs between smooth, non-porous materials, and adsorption is the sole factor in this case.

McBain and Hopkins also state that there is some evidence that the strength of a joint depends on the mechanical properties of the materials and that the thinnest film gives the strongest joint. The strength thus obtained may exceed the strength of the adhesive in mass.

A liquid adsorbed at a solid surface forms a liquid film, and we say that the liquid wets the solid (3).

Osterhof and Bartell (5) distinguish three types of wetting, adhesional wetting, spreading wetting, and immersionsal wetting. Of specific interest is the following: Adhesional wetting occurs when a plane solid surface is lowered parallel to a plane liquid surface until contact is made. They state that the action of adhesives is a practical example of adhesional wetting and that although in the case

of a cemented joint, neither on application nor on removal is the adhesive situated at all times parallel to the solid surface, the final result is the same since either a solid and a liquid interface disappears and a solid-liquid interface appears, or vice versa. Bartell distinguishes between wettability and wetting power. Wettability refers to the solid and represents the tendency of a solid to be wetted and is expressed by the value in ergs of the term $(S_1 - S_1^f)$: (5)

"Where S_1 = Surface tension of solid phase; S_2 = surface tension of liquid phase; S_{12} = interfacial tension between solid and liquid. S_1^f and S_2^f are for the solid and liquid respectively, the free energies remaining in their portion of the solid-liquid interface. The sum of S_1^f and S_2^f then equals S_{12} , the interfacial tension or free energy still resident in the interface."

"This quantity $(S_1 - S_1^f)$ represents the decrease in free energy of a solid surface layer due to partial satisfaction of the unbalanced forces there situated or the extent to which these attractive forces have been neutralized by contact with a liquid. It may thus be regarded as expressing the attraction excited by a solid upon a liquid, and therefore the tendency of the solid to form its portion of a solid-liquid interface."

The formulation of a precise definition of wetting power is more difficult.

"Wetting power represents the tendency of a liquid to wet a solid, but it cannot be as simply expressed as in the former case. Based on similar reasoning to that used for the solid phase, an analogous expression $(S_2 - S_2^f)$ might be selected as a measure of wetting power. This value may be regarded as the actual decrease in free surface energy of a liquid interface when it forms part of a solid-liquid interface. It is the attractive force which a liquid exerts on a solid, and measures therefore its tendency to wet if unhindered by other factors."

Cohesion may be explained in terms of attractive forces operative between unlike charges and between electrical doublets. The linkage

with non-metals is homopolar, the cohesive forces originating from electrical doublets which arise from deformation. The linkage is heteropolar with metals, ionic charges adding to the electrical doublet effect. In polar crystal lattices the units are oppositely charged ions, and the forces of cohesion vary as the inverse square of the distance of separation provided deformation of the electronic shells is absent (22). Broadly speaking, compressibility indicates the nature of the cohesive forces, and hardness and tensile strength may be taken as a quantitative estimate of their magnitude.

Whitesides (23) limits the definition of cohesion in bitumens as the resistance to change of form in either tension or compression and states that since bitumens are liquids approaching finite viscosity at temperatures in which cohesive failures occur, the degree of cohesion becomes largely a function of the viscosity.

APPARATUS

The apparatus of Whitesides (24) was modified and used in testing the adhesive and cohesive strengths of asphalt. A line sketch, showing the necessary dimensions of the breaking apparatus, is given in Figure 1, and a photograph of the apparatus completely set up is shown in Figure 19.

The apparatus consisted of three parts, namely, (1) the test panel, (2) a method of controlling temperature, and (3) a device for breaking the test panels.

The test panel consisted of two steel plates whose polished surfaces were cemented together by a layer of the asphalt to be tested.

The temperature was controlled by circulating water from a constant temperature reservoir through the insulated brass cylinder E (Figure 1) by means of a small gear pump. The temperature was measured by a thermometer placed in the cylinder E and one placed in the constant temperature bath B. The temperature in E and B in no case varied more than $\frac{1}{2}^{\circ}$ Fahrenheit.

The test panel was broken apart by fixing the bottom plate (the concentric ring) and pushing the top circular plate away from it.

The bottom plate of the test panel was fitted into a turned recess on the top surface of the cylinder E and fixed into place by three small clamps. Through the cylinder in a sealed tube passed a plunger P (Figure 1) which was forced up against the top plate of the test panel by a lever L_1 and L_2 . Shot from container S was allowed to fall into a pail at W which forced plunger P up by means of the lever L_1 L_2 .

Figure 1- Line Diagram of Apparatus

Figure 1a-Diagram of Steel Plates
of Test Panel

PROCEDURE

Preparation of Sample

The steel discs are heated in an oven to about 212° Fahrenheit or about 25° above the melting point of the asphalt. The asphalt sample is melted, care being taken to see that no volatile matter is lost, and a layer about 1/16" thick brushed on the bottom plate. The top plate is then taken from the oven and pressed down upon the layer of asphalt, care being taken to see that the asphalt flows out around the bottom edge of the top plate. The sample is then allowed to cool and the excess asphalt scraped off with a hot spatula and then cleaned with solvent.

Method

The water in the inner cylinder B and outer cylindrical jacket C (Figure 1) is heated to slightly higher than the desired temperature and is then circulated by means of a small gear pump D through a brass cylinder E. The test panels, after cooling, are placed in the inner cylinder of the reservoir and allowed to remain 20 to 30 minutes. A panel is then removed and fixed on brass cylinder E (Figure 1) and the bottom plate fixed by 3 clamps. The insulated cover F is placed over the brass cylinder. A force is applied at W on the lever arm L by allowing 1/8" lead shot to fall from S at the rate of approximately 25 pounds per minute (Figure 2). This in turn applies a force through the plunger P on the top plate of A. When the force becomes great enough the sample of asphalt breaks and the amount of lead shot at W is weighed and the strength calculated as shown on page 65.

DATA AND RESULTS

TABLE I
SOURCE OF CRUDE AND PROPERTIES OF ASPHALT SAMPLES

Sample	Manufacturer	Source of Crude*	Softening Point* °F	Penetration* 100 gms/5 sec at 77°F	Ductility at 77°F
A	Standard Oil of La.	Venezuelan (Lagunillas)	166	32	5
B	Standard Oil of La.	Venezuelan (Lagunillas)	194	21	3½
C	Texas Oil Co.	Mixed Texas Crudes	197	33	2½
D	Texas Oil Co.	South American Crudes	197	24	2½
E	Texas Oil Co.	Mixed Texas Crudes	169	36	3½
F	Barber Asphalt Corp.	Texas	196	22	3
G	Barber Asphalt Corp.	Venezuelan (Maracaibo)	163	34	6
H	Barber Asphalt Corp.	Venezuelan (Maracaibo)	195	14	3
I	Barber Asphalt Corp.	Venezuelan (Maracaibo)	162	21	6
J	Shell Oil Co.	Pure Panuco Crude	185	22	6
K	Shell Oil Co.	Pure Sulfur Bluff Crude	175	36	3½
L	Shell Oil Co.	Sulfur Bluff-Venezuelan Blend	187	28	4

*Data as received from manufacturer.

TABLE II

COORDINATES FOR THE RATE OF FLOW CURVE OF LEAD SHOT

Time Seconds	Weight of Lead Shot Pounds
15	5.8
30	11.9
45	18.3
60	25.8
75	30.6
90	38.4
105	43.1
120	46.9
135	52.8

FIG. 2 - RATE OF FLOW CURVE OF LEAD SHOT

TABLE III

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE A

(Venezuelan, Lagumillas)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
10	5 1/2	6.2	5.0
10	3 5/8	4.0	
17.5	16 1/2	18.1	18.9
17.5	16	19.8	
22	39 1/4	43.2	38.3
22	30 1/2	33.5	
36	60	66.0	65.0
36	58 1/3	64.0	
41	59	64.9	65.6
41	58 1/8	63.9	
41	61 5/8	67.8	
50	59 5/8	65.6	63.8
50	60	66.0	
50	54 1/2	60.0	
64	43 5/8	48.0	48.0
66	43 5/8	48.0	48.0
86	37 5/8	41.5	42.4
86	39 3/8	43.3	
86	39 5/8	43.8	
86	37 1/2	41.3	
106	20	22.0	23.5
106	20 3/4	22.9	
106	23 3/4	25.6	
123	15 1/2	17.1	17.4
123	16	17.6	
123	15 3/4	17.4

Figure 3. Adhesion and Cohesion Curve Sample A
(Venezuelan, Lagunillas)

TABLE IV

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE B

(Venezuelan, Lagumillas)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
10	15 7/8	17.5	19.1
10	16 3/8	18.0	
10	20	22.0	
22	21 3/4	23.9	25.3
22	24 1/4	26.7	
36	29 7/8	32.9	33.8
36	30 3/4	33.8	
36	31 1/2	34.7	
50	39	42.8	43.3
50	39 1/4	43.2	
50	40	44.0	
56	45 1/2	50.0	50.0
58	42 3/4	47.1	49.5
58	47 1/4	52.0	
64	44 1/2	49.0	50.0
64	46 3/8	51.0	
69	39	42.9	45.5
69	40 1/4	44.2	
69	42	46.3	
69	44 1/4	48.7	
86	26 3/4	29.5	32.0
86	29 3/4	32.8	
86	30 3/4	33.8	
105	19 1/2	21.5	23.3
105	20 3/8	22.4	
105	23 1/2	25.9	
123	18 1/2	20.4	20.8
123	18 1/4	21.2	
123	19	20.9

Figure 4. Adhesion and Cohesion Curve Sample B
(Venezuelan, Lagunillas)

TABLE V

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE C

(Mixed Texas Crudes)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
10.5	19 5/8	21.6	21.7
10.5	19 3/4	21.7	
22	22 1/2	24.8	26.4
22	24 1/2	27.0	
22	26	27.5	
36	32	35.2	36.3
36	34 1/4	37.7	
50	36 1/4	38.8	41.9
50	37 1/8	40.8	
50	42	46.2	
60	43 1/2	47.9	49.7
60	45	49.5	
60	47	51.7	
77	36	39.6	41.8
77	38 1/2	42.4	
77	39 1/2	43.5	
86	30	33.0	35.8
86	31 1/2	34.7	
105	21 1/2	23.7	25.9
105	22 1/2	24.8	
105	26 1/2	29.2	
123	18	19.8	20.6
123	18 5/8	20.5	
123	19 5/8	21.6

Figure 5. Adhesion and Cohesion Curve Sample C
(Mixed Texas Crudes)

TABLE VI

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE D

(South American Crudes)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
10.5	10 5/8	11.7	12.8
10.5	11 1/2	12.7	
10.5	12 7/8	14.1	
22	25 1/4	27.8	27.8
36	34 1/8	37.6	38.0
36	34 7/8	38.4	
50	52 1/8	57.3	57.6
50	45 7/8	50.5	
50	59 3/8	65.2	
60	54 1/2	60.0	62.5
60	59	64.9	
70	48 1/2	53.4	55.7
70	50	55.0	
70	53 1/2	58.9	
86	30 1/4	33.3	37.6
86	33	37.2	
86	35 3/4	39.4	
86	36 5/8	40.3	
105	19	20.9	27.3
105	26 1/2	29.2	
105	29	31.9	
123	15 1/2	17.1	18.5
123	16	17.6	
123	19	20.9

Figure 6. Adhesion and Cohesion Curve Sample D
(South American Crudes)

TABLE VII

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE E

(Mixed Texas Grades)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
10.5	9 1/2	10.4	13.6
10.5	11 1/4	12.4	
10.5	16 5/8	18.0	
17.5	29 1/2	32.5	24.7
17.5	31 1/4	34.2	
17.5	34 1/4	37.6	
26	36 1/4	59.9	41.4
26	39	42.9	
36	59 1/4	65.3	65.4
36	59 1/4	65.3	
36	59 1/2	65.5	
40	59	64.9	66.9
40	60	66.0	
40	63 1/2	69.9	
48	58	63.8	64.6
48	58 1/2	64.4	
48	59 1/4	65.3	
68	36 5/8	40.4	43.3
68	39 5/8	43.7	
68	41 5/8	45.8	
77	31 5/4	33.8	35.3
77	31 1/4	34.4	
77	32 1/2	35.8	
77	33 3/4	37.2	
86	29 1/8	32.0	33.4
86	30 5/8	33.7	
86	31 3/8	34.6	
106	20 1/2	22.6	24.7
106	21 1/4	23.4	
106	25 1/2	26.1	
123	15 1/2	17.1	19.0
123	17 1/8	18.9	
123	19 1/4	21.1

Figure 7. Adhesion and Cohesion Curve Sample E
(Mixed Texas Crudes)

TABLE VIII

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE F

(Texas Crude)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
17.5	9 1/4	9.9	
17.5	11 1/8	12.3	11.8
17.5	12	13.2	
36	19 5/8	21.6	
36	24	26.4	24.0
48	25	27.5	
48	30 1/2	33.5	31.8
48	31 1/2	34.6	
68	42 3/8	46.6	
68	43 1/8	47.5	47.3
68	43 5/8	47.6	
77	45 1/2	50.0	
77	46 1/4	50.9	52.3
77	50 1/2	55.6	
86	39	42.9	
86	40 5/4	44.8	45.3
86	43 1/8	48.3	
105	24 5/8	27.1	
105	25 7/8	28.5	28.1
105	26	28.6	
123	19	20.9	
123	19 1/2	21.5	21.6
123	20 3/8	22.4

Figure 8. Adhesion and Cohesion Curve Sample F
(Texas Crudes)

TABLE IX

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE G

(Venezuelan, Maracaibo)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
10.5	8 5/8	9.5	12.0
10.5	9 3/4	10.7	
10.5	14 1/2	15.9	
17.5	16 3/4	18.4	26.7
17.5	27 1/4	30.0	
17.5	28 7/8	31.8	
36	36	39.6	47.5
36	44 1/2	49.0	
36	48 7/8	53.8	
48	52 1/2	57.8	62.1
48	58 3/8	64.3	
48	58 3/8	64.3	
62	46 1/2	51.2	53.9
62	51 1/2	56.7	
68	47	51.8	54.3
68	49 1/2	54.5	
68	51 1/2	56.7	
86	31 1/4	34.4	36.1
86	33 5/8	37.0	
86	33 5/8	37.0	
105	21 3/4	23.5	24.6
105	22 1/8	24.4	
105	23 3/4	26.2	
123	16 1/4	17.9	19.8
123	18 5/8	20.5	
123	19 1/8	21.1

Figure 9. Adhesion and Cohesion Curve Sample G
(Venezuelan Maracaibo)

TABLE I

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE H

(Venezuelan, Maracaibo)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
18	3 5/8	4.0	4.0
20	6 3/8	7.0	8.5
20	9	10.0	
36	9 3/4	10.7	12.5
36	13 1/8	14.5	
48	15 1/4	16.8	20.1
48	17 3/4	19.5	
48	21 3/4	23.9	
66	27	29.7	29.7
69	30 5/8	33.7	34.9
69	32 7/8	36.2	
88	35 3/4	39.4	51.2
88	49 1/2	54.5	
88	54 1/4	59.7	
106	26 7/8	29.6	33.0
106	26 3/4	31.6	
106	34 3/8	37.8	
123	19 1/4	21.2	23.5
123	20 1/4	22.3	
123	24 1/2	26.9

Figure 10. Adhesion and Cohesion Curve Sample H
(Venezuelan, Maracaibo)

TABLE XI

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE I

(Venezuelan, Maracaibo)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
8.5	5	5.5	8.8
8.5	5	5.5	
8.5	14 1/8	15.5	
15.5	9 1/4	10.2	13.6
15.5	15 1/2	17.0	
37	24 7/8	27.4	28.9
37	26 1/8	28.7	
37	27 7/8	30.7	
49	43 1/4	47.6	52.0
49	51 1/4	55.5	
50	49	53.9	55.0
50	51	56.1	
58	54 1/2	60.0	61.1
58	55 1/2	62.2	
60	59	64.9	64.9
68	49	53.9	57.2
68	51 3/4	57.0	
68	55 1/4	60.7	
85	41	45.1	46.2
85	43	47.3	
105	19 7/8	21.8	23.0
105	20 5/8	22.7	
105	22 3/8	24.6	
119	16	17.6	19.1
119	17 1/4	19.0	
119	19	20.9

Figure 11. Adhesion and Cohesion Curve Sample I
(Venezuelan, Maracaibo)

TABLE XII

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE J

(Pure Pantuco Crudes)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
8.5	9	9.9	10.8
8.5	10 5/8	11.7	
18.5	8 5/8	9.5	11.8
18.5	9 1/8	10.1	
18.5	14 1/2	15.9	
32	25 1/2	28.0	29.0
32	27 1/4	30.0	
37	31 1/4	34.4	35.4
37	32 1/4	35.5	
37	33	36.3	
50	43	47.3	52.9
50	44	48.4	
50	57 1/4	63.0	
59	49 1/2	54.5	55.6
59	52	57.2	
68	41 5/8	45.5	48.0
68	45	49.5	
68	44 1/2	49.0	
85	36	39.6	40.1
85	36 1/2	40.1	
85	37	40.7	
105	22 7/8	25.2	26.0
105	23 7/8	25.4	
105	25	27.5	
119	17 1/2	19.3	20.6
119	18 1/4	20.1	
119	20 1/4	22.2

Figure 12. Adhesion and Cohesion Curve Sample J
(Pure Panuco Crude)

TABLE XIII

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE K

(Pure Sulfur Bluff Grade)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
8.5	13	14.5	15.3
8.5	13 1/2	14.8	
8.5	15 1/2	17.0	
15.5	24 3/4	26.2	31.7
15.5	30 3/4	33.7	
15.5	32 1/4	35.4	
26	32	35.2	38.9
26	36 1/2	40.2	
26	37 1/2	41.3	
37	45 1/2	50.1	54.2
37	50 7/8	56.0	
37	51 1/2	56.7	
48	49 1/2	54.5	56.1
48	52 1/2	57.8	
50	46 1/4	50.0	51.3
50	47	51.8	
68	37	40.7	43.7
68	38	41.8	
68	44 1/4	48.7	
86	29 3/8	32.5	35.7
86	32 3/8	36.7	
86	34 5/8	38.2	
105	17	18.7	21.1
105	19 3/8	21.3	
105	21 1/4	23.4	
119	17	18.7	19.0
119	17 5/8	19.4	
121	15 5/8	17.2	17.2

Strength - Pounds per Square Inch

80

60

40

20

0

0

20

40

60

80

100

120

140

Temperature - Degrees Fahrenheit

Figure 13. Adhesion and Cohesion Curve Sample K
(Pure Sulfur Bluff Crude)

TABLE XIV

RESULTS OF ADHESIVE AND COHESIVE TESTS OF SAMPLE L
(Sulfur Bluff-Venezuelan Blend)

Temperature °Fahrenheit	Weight of Lead Shot Pounds	Strength Pounds/square inch	Average Strength Pounds/square inch
8.5	8 1/4	9.1	10.7
8.5	9 1/2	10.5	
8.5	11 1/4	12.4	
15.5	11	12.1	14.9
15.5	12 3/4	14.1	
15.5	17	18.7	
37	39 3/4	43.7	45.7
37	40 3/4	44.8	
37	44 1/4	48.7	
50	49	53.9	58.2
50	56 7/8	62.6	
58	54	59.4	61.7
58	55 1/2	61.0	
58	59	64.9	
68	47 3/4	52.5	53.5
68	48	52.8	
68	50 3/8	55.4	
86	34 3/8	37.8	39.8
86	36 3/4	40.4	
86	37 3/8	41.1	
105	21 3/8	23.5	24.0
105	21 5/8	23.8	
105	22 1/2	24.8	
119	16 1/2	18.2	19.6
119	18 1/8	19.9	
119	18 5/4	20.6

Figure 14. Adhesion and Cohesion Curve Sample L
(Sulfur Bluff-Venezuelan Blend)

PHYSICAL PROPERTIES

SOFTENING POINT

The Ring and Ball method (1) of testing for softening point was used in this investigation. This method has been standardized by the American Society for Testing Materials.

"The softening of bituminous materials generally takes place at no definite moment or temperature. As the temperature rises, they gradually change from a brittle or exceedingly thick and slowly-flowing material to a softer and less viscous liquid. For this reason the determination of the softening point must be made by a fixed, arbitrary and closely defined method if the results are to be comparable." (1)

The apparatus consisted of the following:

- (a) A brass ring $5/8$ inch in inside diameter, $23/32$ inch outside diameter, and $1/4$ inch deep.
- (b) A steel ball $3/8$ inch in diameter weighing between 3.45 and 3.55 grams.
- (c) An 800 c.c. glass beaker, low form.
- (d) A 30° to 180° Fahrenheit thermometer.

The sample was melted, stirred thoroughly, and poured into the brass ring so that when it was cooled an excess was left. The ring rested on an amalgamated brass plate to prevent the asphalt from adhering to it. When the sample was cooled the excess material was cut off cleanly with a heated spatula.

For those asphalts fusing below 176° Fahrenheit the glass beaker was filled to a depth of $3\frac{1}{4}$ inches with freshly distilled water at about 70° Fahrenheit (1). The ring containing the sample was suspended in the water so that the lower surface of the filled ring was 1 inch above the bottom of the beaker and its upper surface was 2 inches

below the surface of the water. The ball was placed in the water at the same time but not on the sample. The thermometer was suspended so that the bottom of the bulb was level with the bottom of the ring. The ball and ring were allowed to remain in the water for fifteen minutes. The ball was then placed in the center of the upper surface of the asphalt in the ring and the water heated by means of a small Bunsen flame.

The heat was applied in such a manner that the temperature of the water was raised $9^{\circ}\text{F.} (+ .9^{\circ}\text{F.})$ each minute. At the instant the asphalt touched the bottom of the beaker the temperature was recorded and reported as the softening point.

For those asphalts fusing above $176^{\circ}\text{Fahrenheit}$ the method given above was employed, except that U. S. P. glycerin was used instead of water.

The softening points of the asphalt samples used in this investigation are given in Table IV.

TABLE XV

SOFTENING POINTS OF ASPHALT SAMPLES

Sample	Temperature °Fahrenheit
A (Venezuelan, Lagunillas)	190
B (Venezuelan, Lagunillas)	176
C (Mixed Texas Crudes)	200
D (South American Crudes)	201
E (Mixed Texas Crudes)	171
F (Texas Crudes)	201
G (Venezuelan, Maracaibo)	168
H (Venezuelan, Maracaibo)	193
I (Venezuelan, Maracaibo)	162.5
J (Pure Pemaco Crude)	199
K (Pure Sulfur Bluff Crude)	194
L (Sulfur Bluff-Venezuelan Blend)	188

PENETRATION

"Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time and temperature." (1)

A sketch of the penetrometer showing the essential parts is given in Figure 15. The base A may be leveled by thumb-screws B and is attached to the standard C and also the platen D on which the sample is placed. The standard C carries an adjustable bracket F and also the dial T, which measures the penetration in one-tenth millimeter, by a rack on a sliding gauge L. The plunger O acts as a brake, which holds the needle bar with load W. When it is pressed inward the plunger permits the needle and weight to act upon the test sample without friction.

The container for holding the asphalt to be tested was a flat-bottomed, cylindrical dish $2 \frac{3}{16}$ inches in diameter and $1 \frac{3}{8}$ inches deep. A hardened and highly polished needle, approximately 2 inches long and with a diameter of 1.00 to 1.02 mm., was used in this test. It was symmetrically tapered at one end to a cone approximately $\frac{1}{4}$ inch in height and had an angle within the range $8^{\circ}40'$ and $9^{\circ}40'$. The tapered end was of the shape of a truncated cone, the smaller base of which was from 0.14 mm. to 0.16 mm. in diameter.

The sample of asphalt was completely melted at the lowest possible temperature and stirred thoroughly until it was free from air bubbles. It was then poured into the container to a depth of about $\frac{5}{8}$ inch. After an hour the sample was placed in a water bath maintained at the

Figure 15- Sketch of Penetrometer Showing Essential Parts.

desired temperature of testing and allowed to remain for one hour.

The sample was placed in a small transfer dish filled with water from the water bath. The transfer dish containing the sample was then placed on the stand of the penetrometer.

The needle loaded with 200 grams was adjusted to make contact with the surface of the asphalt. The dial was brought to zero reading. The needle was released for 5 seconds, after which the depth of penetration was measured by the rack on the sliding gauge L (Figure 15).

The penetrations reported in Table XVI are the average of three tests whose values varied less than four points from minimum to maximum.

TABLE XVI

PENETRATION OF ASPHALT SAMPLES AT VARYING TEMPERATURES

Sample	Penetration 200 gms./5 sec. at 38°F	Penetration 200 gms./5 sec. at 58°F	Penetration 200 gms./5 sec. at 77°F	Penetration 200 gms./5 sec. at 100°F
A (Venezuelan, Lagunillas)	10.5	17.5	26.5	46
B (Venezuelan, Lagunillas)	14	22	36	64.5
C (Mixed Texas Crudes)	18	25.5	36	56
D (South American Crudes)	11	19	26	45
E (Mixed Texas Crudes)	17	25.5	39.5	64
F (Texas Crudes)	10.5	17	26.5	45
G (Venezuelan, Maracaibo)	16.5	25	39.5	66
H (Venezuelan, Maracaibo)	5	9.5	16	29.5
I (Venezuelan, Maracaibo)	8	15	26	52.5
J (Pure Pannoo Crude)	11.5	20	31	54
K (Pure Sulfur Bluff Crude)	15	26	42.5	62
L (Sulfur Bluff-Venezuelan Blend)	11	18.5	31.5	54.5

Figure 16. Penetrations of Asphalts. Samples A, B, E, L.

Sample A-(Venezuelan, Lagunillas)

Sample B-(Venezuelan, Lagunillas)

Sample E-(Texas Crude)

Sample L-(Sulfur Bluff-Venezuelan Blend)

Figure 17. Penetrations of Asphalts. Samples C, D, F, G.

Sample C - (Mixed Texas Crudes)
 Sample D - (South American Crudes)
 Sample F - (Texas Crude)
 Sample G - (Venezuelan, Maracaibo)

Figure 18. Penetrations of Asphalts. Samples H, I, J, K.

Sample H- (Venezuelan, Maracaibo)
 Sample I- (Venezuelan, Maracaibo)
 Sample J- (Pure Panuco Crude)
 Sample K- (Pure Sulfur Bluff Crude)

DISCUSSION OF RESULTS

A close similarity was observed between the shapes of the experimental, strength-temperature curves for all asphalts tested. The total curve in each instance was composed of two separate curves, one adhesion and the other cohesion.

Theoretically the adhesion, curve X (Figure 4), increases with increasing temperature, which is probably caused by better adhesional surface wetting as suggested by McBain and Hopkins (14) and Bancroft (3). Also, the cohesion, curve Y (Figure 4), increases with decreasing temperature until it reaches a maximum, which is generally coincident with the temperature at which the ductility is zero (1).

The intersection of these two theoretical curves gives a point P (Figure 4) at which the adhesive and cohesive strengths are equal at the same temperature. This point shall be referred to as the critical point.

In actual practice this point would be considered as the maximum working strength because at any temperature below the critical point internal cohesion becomes greater than adhesion and failure would occur in adhesion first. Similarly, at temperatures above the critical point adhesion is greater than cohesion; therefore, failure would occur in cohesion.

In this investigation, however, the maximum strength did not reach the critical point, and the curves rounded off slightly below this point. This was caused by the sample breaking in a combination of adhesion and cohesion in the region near the critical point (Figure 20). In order to reach this point the samples would have to break in adhesion alone at any temperature up to the critical point and in cohesion

at any temperature above it.

It was observed that the faster the rate of loading the more nearly the maximum strength reached the intersection of the theoretical curves. Thus it appears that in order to obtain the true maximum strength the load would have to be applied at a rate such that failure of the sample occurs instantaneously on application of load.

From an observation of the experimental data, there was no correlation of adhesion or cohesion with the commonly accepted tests, penetration and softening point. Neither did the ductility data as received from the manufacturer give any correlation. Therefore, the best method of determining the quality or adaptability of an asphalt for cementing purposes would be a direct measurement of adhesive and cohesive strengths as carried out in this investigation.

SUMMARY AND CONCLUSIONS

When the working conditions of an asphalt cement are specified for any application the area inclosed by the temperature limits and the desired strength must fall under the strength-temperature curve; otherwise, the asphalt will fail in tension. In those cases where the necessary working conditions are known this test could be used in writing specifications for asphalt cements.

APPENDIX

Sample Calculations

In order to change pounds of lead shot at W (Figure 1) to strength of asphalt sample in pounds per square inch, the force downward at W was changed to force upward at P by use of moments. This value of force P was divided by area of asphalt layer on test panels to obtain strength in pounds per square inch.

Area covered by asphalt sample (from Figure 2) -

$$A = \frac{\pi [(29/16)^2 - (13/16)^2]}{4} = 4.64 \text{ square inches}$$

Force at P (from Figure 1) -

$$W \times 17 \frac{1}{4} = P \times 3 \frac{3}{8}$$

$$P = \frac{17 \frac{1}{4} W}{3 \frac{3}{8}} = 5.111 W$$

Strength in pounds per square inch -

$$S = \frac{P}{A} = \frac{5.111 W}{4.64} = 1.10 W$$

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Figure 19. Photograph of Apparatus.

Figure 20. Test Panels after Break.
(A) Break in Cohesion.
(B) Break around Region
of Critical Point.
(C) Break in Adhesion.

The Author wishes to express his sincere appreciation to Mr. George W. Whitesides for his helpful suggestions and to Kentucky Celar and Chemical Company for most of the materials used in this investigation.

VITA

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The author, Harold Rose, son of James M. and Margaret Rose, was born August 11, 1918, in Louisville, Kentucky. He obtained a primary education in the Louisville Public Schools and a secondary education at Louisville Male High School. After graduating from High School in June, 1935, he entered the Speed Scientific School, University of Louisville, in September, 1935. He received a degree of Bachelor of Chemical Engineering in June, 1939, and a degree of Master of Chemical Engineering in June, 1940.

Industrial experience was obtained at the following:

1. Cohert Refractories Co., 16th & Lee, Louisville, Ky.
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2. Henry Vogt Machine Co., 10th & Ormsby, Louisville, Ky.
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3. Louisville Water Co., Frankfort & Zorn Aves., Louisville, Ky., 3 months, 1937.
4. Seagram's Distilleries, 7th St. Rd., Louisville, Ky.
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5. Kentucky Color & Chemical Co., 34th & Bank, Louisville, Ky., 14 months, 1939-40.

While an undergraduate he played three years of varsity football and was elected to the Sigma Tau, honorary engineering fraternity.