THE ACCELERATED WEATHERING
OF
LIQUID ASPHALTIC OILS

Thesis for the Degree of B. S.
Norman W. Sageman
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THESIS

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of
Liquid Asphaltic Oils

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by

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* * *

INTRODUCTION

Since modern civilization is founded on transportation and communication, the highway offers a very essential means of transportation. United States today has over 3 million miles of roads of which approximately 2 million miles is of the unimproved type. There still is a large mileage of roads to be improved, so more and better methods of building, and materials for building, can be found and applied to improving the roads.

The performing of this thesis will do a lot to develop the technique, judgment, and experience of the writer on the subject of asphaltic cements besides contributing something new to highway research work, and, at the same time, this is an excellent opportunity to show some definite relationships between various weathering tests.

The object of this thesis is to correlate weathering of liquid asphaltic cements with their quality, to develop a suitable quality test that can be used to distinguish between good and poor asphaltic oils, a test that can be easily and quickly carried out in the laboratory with the least amount of equipment and with very little special training on the part of the operator.

History of Weathering

In the early days of photography, solutions containing asphalts with some ethereal oils were used for preparing the sensitive photographic film. On exposure to light, air and moisture under the lens of a camera, certain changes were found to take place in the asphaltic coating because when the film was subjected to the action of certain solvents, the portion which had been in contact with the light became insoluble, whereas the portions protected from the light readily dissolved in the solvent. The first real experimenting with different asphaltic materials in the photographic industry was carried as early as 1816 by Joseph Niepce by rather crude methods as illustrated above.

It was soon observed that certain forms of asphalts were more sensitive than others, and Syrian native asphalt became very popular on account of its purity, solubility, and sensitiveness to the lights' rays[1]. By the addition of the chemical compound, sulfur chloride, to the native asphalts,[2] the sensitivity of them toward light was greatly increased, but the petroleum asphalts were practically rendered inert in its presence. Still further investigations showed that petroleum asphalts free from paraffine were relatively the most sensitive toward light.[3]

Another industry where the weather-resisting properties of bituminous substances are of primary importance is in the manufacture of paints, varnishes, and enamels. Mr. Toch noted that bituminous materials on exposure to sunlight decomposed with the liberation of "free" carbon.[4] His experiments also indicated that this action was inhibited by incorporating an opaque pigment. He also pointed out that animal and vegetable oils are not affected in this manner, and when blended with bituminous materials, apparently retard the action.

Mr. Hubbard and Mr. Reeve[5] exposed several samples of asphalts to the weather under cover for a period of one year. At the end of the test the residues showed an increase in weight and in hardness.

They concluded that the hardness of all bitumens is not due to the loss of lighter constituents alone, it may be due to the action of light and oxidation. It may be caused by physical phenomena, such as loss of lighter oils by volatilization, or mechanical changes such as rearrangement, inter-reaction, or oxidation of certain constituents.

In continuation of the above work, Mr. Reeve with the aid of Mr. Lewis[6] weathered several more samples in panels under glass and the results noted. Results were compared with the loss on heating test on the corresponding materials. It was found that the action of weathering was greater than the loss on heating at 163°C although comparable. It was decided that all bitumens when exposed to weather undergo changes that are due to something more than the loss of volatile matter and they may also absorb some oxygen.

Research work continued to be carried on with the various bituminous materials along the line of weathering and artificial weathering. The changes brought about by exposure to the elements are quite complicated and involve one or more of the following reactions[7]:

Evaporation: This represents the gradual loss of volatile constituents on exposure to air and the sun's heat. With any bituminous substance, the rate of evaporation depends almost entirely upon the temperature, the higher the temperature the greater will be the amount of evaporation providing all other things are kept equal. The determination of volatile matter is usually regarded to be an accelerated evaporation test, which is supposed to show in a relatively short time at an elevated temperature what takes place over a lengthy period when exposed to the elements of nature. This is not, however, true as will be explained later.

Oxidation: This takes place on exposure to air and progresses more rapidly at high than at low temperatures. The effect of oxidation is twofold, and involves the direct union of oxygen with the bituminous substances, also the elimination of a portion of the hydrogen or carbon in the form of water or carbon dioxide. The absorption of oxygen is accompanied by a gain in weight whereas the elimination of hydrogen or carbon is

accompanied by a loss in weight. The extent and nature of these reactions will depend upon the substance itself and also on the conditions to which it is subjected.

<u>Carbonization</u>: This represents the formation of "free" carbon in the bituminous material and is induced by an extensive elimination of hydrogen to an extreme. The deposits of free carbon generally contain a small percentage of hydrogen so are not composed of free carbon completely. This reaction progresses quite rapidly in sunlight and upon subjecting the bituminous material to high temperatures.

<u>Polymerization</u>: This is due to a condensation of the molecules and manifests itself by a hardening or setting of the substance. It usually occurs to a greater extent on the surface of a material than it does beneath. It seems that polymerization takes place in bituminous materials, if they are allowed to stand for some time besides also taking place to a greater or lesser extent upon heating to a high temperature. Polymerization is extremely noticeable in fatty-acid pitches.

<u>Effect of Moisture</u>: Bituminous substances will absorb water or the water will leach out the soluble constituents. Thus oxidation intensifies this action because oxygenated substances have a greater affinity for moisture.

With some further study into the chemistry of bituminous materials it is an apparent fact that they are very complicated structures and consist of mixtures of numerous chemical substances in varying proportions.[8] None of the commercial products have definite composition but may consist of saturated hydrocarbons and unsaturated hydrocarbons in various proportions.

Hydrocarbons in which the adjacent carbon atoms are joined by one

valences are considered saturated. These compounds are quite stable and will not undergo much chemical changes.

Unsaturated hydrocarbons are those in which some of the adjacent carbon atoms are joined by two or three valences. This type of compounds is less stable as the extra bonds tend to form new saturated molecules in the presence of chemically active compounds.

Research is being carried on by the Bureau of Public Roads on liquid asphaltic road materials in continuation of their work on Slow-Curing types[9] and later the medium and rapid-curing types[10] of liquid asphaltic materials. The latest work undertaken by them is the testing of road tars.[11]

In the first of the above three mentioned works it was shown that the action of sunlight, heat, and air on the materials when they were exposed in thin films produced residues with physical and chemical characteristics differing greatly from those of the residues obtained from the usual laboratory heat tests. It was also shown when these materials were mixed with a standard sand, molded into cylinders by the Hubbard-Field method, and subjected to the same exposure as the thin films the stability developed by each could not be attributed to the loss in volatile matter. In other words this method of testing cannot be used to determine the differences in stability. The hardening of the materials occur greater upon exposure than during distillation due to the causes other than the loss of volatile matter.

From the results of the tests on medium - and rapid - curing oils it was found that the Oliensis Spot test is more sensitive than the microscopic test in the detection of materials that have been overheated during the process of manufacture. Although neither test seems definitely to be able to distinguish products that willweather badly. Also it was found that

carbonization occurred in some of the oils in both the laboratory and exposure conditions that had a very high solubility in carbon disulphide and carbon tetrachloride.

Most all of the above conclusions were verified in the tests on tars and the following conclusions were also drawn: (1) the bond strength developed by exposure was far greater than that obtained from loss of just the volatile matter. (2) the ultimate loss on exposure approximates the total loss of volatile matter in the standard distillation test. (3) in general the index of free carbon in the residues from exposure is higher for the tars that had initially a low free carbon content and also with a much increased hardness of the exposure residues.

At present many of the laboratory heat tests are believed to produce conditions more severe and dissimilar to service conditions. The past investigations have shown in some cases that physical and chemical characteristics are developed upon exposure that satisfactorily withstand the standard laboratory specifications tests.

The standard tests for acceptable materials restrict the materials to a limited number of sources or manufacturing processes without anything to show the weather-resisting properties or life of the material thus obtained. This leads the author to believe that some modification or modifications of the present laboratory heat tests so that the residues developed may be recognized as a means for telling the good life of a bituminous substance.

The writer intends to take a series of various asphaltic road oils of the type SC-4A. and subject them to various heat tests and exposure tests. The results of these tests will be correlated together by making various tests on the residues from each test.

The main test will be the bromine-absorption test on all the residues as well as the original materials. Since Bromine is active chemically, and the road oils being tested are in the classification of unsaturated hydrocarbons[12] that are most easily oxidised, reduced, condensed, and halogenated, some comparable results ought to be obtained.

Air, light, moisture, and temperature initiate and promote organic reactions.[12] Therefore with the above considerations it seems that determinations of unsaturates could serve as a measure of weathering resisting characteristics of bituminous materials.

PROCEDURE

The most of the materials necessary to work with were already in the highway laboratory. The different oils used were obtained from samples that the Michigan State Highway Department had obtained from the various refining companies. The highway department also furnished the plates for the weather-ometer test and the necessary chemical compounds used during the experimenting. All of the other equipment and materials used were in the college highway laboratory except the arc lamp weather-ometer.

The weather-ometer was built similar to the type used by other research laboratories.[13] The carbon arc lamp was borrowed from the electrical engineering department of the college. It was so fixed up that it operated on the college 110 v. to 130 volt alternating current circuit.

The stand or base had to be constructed from materials received from college stores and repair shop. For the legs 2¼" cast iron pipe were used and the top of table was made from 1" angle iron stock. First the legs were cut at a length of 36" and holes drilled in them to attach the angle iron top and braces to them. The top outside angles iron was made square being 30" on a side which was the necessary size needed in order that the test plates could be placed far enough from the arc lamp. The top was attached to the legs and an angle iron brace was placed around the legs and fastened at a distance of 22" from the top of the stand. Eight small angle irons were fastened to a center gusset plate and then placed on top of the stand from where their lengths were marked to be cut off at the correct length to form the base for the two circular rings, one 29" in diameter and the other 20" in diameter, that were to support the test plates. This base was then fastened to the stand and the two

rings made from the angle irons fastened to it by means of ¼" bolts. It was necessary to put shims under some parts of the circular rings in order to have them perfectly level because the type of oils tested are quite viscous so in order to have the oil remain on the plates in a uniform thickness it was necessary to have them perfectly level. The support for the arch lamp consisted of two ¾" pipes 6 feet long connected by elbows to a pipe of the same size 30 inches long. The short pipe was the cross-bar and the two long pipes the legs that were fastened to the top angle iron of the stand and also the angle iron brace that was placed down 22 inches from the top of the stand.

In order to keep the dust off of the plates and to keep the air drafts from affecting the arc lamp it was necessary to enclose the sides and bottom of the weather-ometer with heavy building board material, with the top being covered with a large piece of cloth to keep the dust out and also to allow some ventilation to carry off the gases and vapors that were formed.

The next thing was to put an even film of each oil upon the aluminium test plates which were 2¾" by 5 7/8". The method of putting an even film on the plates by means of the instrument called the asphalt trimmer. This trimmer consists of a stationary metal table and an electrically heated cylindrical element with vertical adjustments which permits the plates to be drawn through under the element and an even thickness of oil is left on the plate. A machine like this would be too expensive to buy so it was necessary to use the method of centrifugal force to spread an even layer of oil on the plates.

An old centrifugal force milk testing apparatus was obtained from the dairy department and it was remodeled so the test plates could be fastened

on a flat brass plate directed over the spindle and after a drop of oil was placed on the plate the crank was turned at approximately 100 revolutions per minute for about three minutes. It was found that the film thickness to be deposited on the plate, if the plate is kept at a temperature of 125°F during the spreading of the oil, to be approximately 0.001 cm. This thickness was calculated from the weight of the oil being deposited on the plate and the area of the plate being known.

After several trials were made to get the most uniform film upon the plates, the 16 aluminium plates were marked and weighed, the weights being recorded. Then a film of each oil was placed upon the plates by means of the centrifugal force apparatus that was kept at a temperature of 125°F and turned at a speed of 100 r.p.m. for three minutes. After all the plates with the oil on them were again weighed and their weights recorded, they were placed around in a circular fashion on the rings in the arc lamp weather-ometer.

The date and time of turning on the arc lamp was recorded after which an inspection of the plates was made every three hours. Any changes in their appearances was noted as well as the temperature within the weather-ometer. The arc lamp was kept running continuously and the plates were rotated about under the arc lamp so each would receive the same amount of light and heat from the arc. The results of this continuous test will be given in the data and results of the experimenting later in this report. To have more results to correlate with the continuous running test, samples of the same oils were run under the arc lamp in aluminium plates according to the modified accelerated weathering test with film thickness of .025 inch until failure. This modified weathering test[14] is intended to imitate and intensify the action of sun, rain, and low temperatures upon bituminous

materials in the temperature zone. It is generally conceived that 30 cycles of 24 hours is equivalent to 1 year of actual exposure outside. The cycle consists of 18 hours under the arc light at 140°C then the samples were placed in a water bath for 5 hours at room temperature. One hour was allowed for inspection then the plates were placed under the arc lamp for another cycle. Refrigeration was applied to the plates for a period of 1 hour three times a week. The samples were run until failure and then the samples were tested by the bromine absorption by the weathered oils.

By testing the water from the water bath that the samples were placed in, it was found that it showed an acid reaction which is an indication that during the weathering process chemical changes take place so that the part becoming soluble in water is acid in character. The compounds in the water were found to be aldehydes by testing the solution with silver nitrate and the result was a silver precipitate formed.

Heating in the Oven: One gram of each oil was weighed out on the analytic balance into small circular tin pans that were 2¾" in diameter. This was given a film thickness of .026" of oil in each pan.

After the oven, which was heating by electric heating coils, was brought up to 143°C the automatic rheostat on the oven was adjusted so the temperature would be kept constant at all times at 143°C.

The 16 samples were run in the oven at one time. Every three hours a visual inspection was given them to note any change in characteristics and to note the time that each oil lost its adhesiveness or life. That is to say when the oil became so hard or broken up that it would be of no value as far as being of any use as a binding material. The results will be found tabulated in the data later in this report.

After all the oils had failed, the pans were taken out of the oven and accurately weighed on an analytic balance. From the original weight and final weight the loss in weight during the test was found. All the samples were then tested for the amount of Bromine they would absorb, and the Bromine - absorption value of the oils after heating determined.

Heating in Oven, Ottawa Sand with 3 Percent Film of Oil on It.

A 3 percent film of oil was chosen as the approximate amount of oil that would cover particles of aggregate in the construction of an oil road. Next it was necessary to determine what amount of oil to add to a certain amount of sand in order to get a film coat on the ottawa sand of the correct thickness. Ottawa sand was used because the particles are more nearly of a uniform size than other aggregate materials.

A sample of the sand was washed and screened through a 20 mesh screen marked .0328 inches close fit. One gram of the material was taken and thoroughly mixed from which a number of samples were taken and the number

of kernels lying close together between two marks on a flat piece of paper 1 cm apart was found from a number of trials. The average obtained was 12 kernels from which the diameter of one particle was obtained.

The surface area of the particles was obtained under the assumption that the particles are spheres. From the counting of 0.1 gram samples it was determined that 1 gram contained 1400 particles of sand from which the total surface area of one gram of the sand was computed. Computations were computed for a 500 gm sample of sand and a 3% film of oil, and it was found that the thickness of a 3% oil film was approximately 0.00046 inch.

It was necessary for the bromine absorption test to have ½ gram of oil so the amount of sand and oil necessary for the test was computed and mixed together. In order to get the sand all well coated it was necessary to heat the mixture and stir it up well with a spatula until it was well mixed.

A circular tin pan 3½" in diameter was used to put the sample of oil and sand in as they give a thickness of material in the pan sufficient so a change in the adhesiveness or life of the the oil to hold the particles could be noted upon inspection. The weight of the pan, the weight of the pan and sample were determined on an analytic balance.

Eight samples were tested in the oven at one time. The samples were inspected every three hours and when the oil lost its tackiness or had failed, it was taken out of the oven and the time it took the sample to fail was recorded. The sample was weighed and the loss in weight determined. After the first eight samples had failed then the other eight samples were run until failure. The test in the oven was run at a temperature of 143°C the same as in heating the oils in the tin pans. From the samples the oil was removed from the sand by the solvent, carbon disulphide and the bromine absorption test run on each sample.

Oxidation by Air: The author will not go into any detail about this test as it is the object of Mr. Moore's thesis. The author of this thesis obtained samples of the residue for each oil after it had air blown through it for two hours at the rate of 500 cc per minute at a temperature of 180°C to run the bromine absorption test on them so they could be compared to the results for the other weathering tests. The only difference in comparing the results is the fact that the loss in weight of the original oils were not determined during the air blown test so the bromine absorption value can only be computed on the weight of the residue while with the other tests completed the bromine absorption value was computed for 100 gms of the original oil.

The Bromine Absorption Test[15]

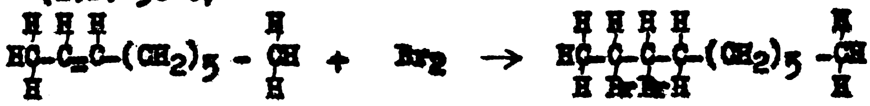
Since bromine, chlorine or iodine are used to test the unsaturatedness of hydrocarbons, it was decided by the author to use the bromine absorption to correlate the results of the different weathering tests together. The speed of the addition reaction of olefinic hydrocarbons is generally rather high with bromine so the test can be used without taking much time. It may be well to give a few typical reactions of unsaturated straight chain hydrocarbons upon treatment with bromine before giving the procedure used in performing the absorption test.[16]

(1) Compounds with one double bond.

$$H-CH_2-CH=CH-CH_2-CH_3 + Br_2 \rightarrow HCH_2-CH-CHBr-CHBr-CH_3$$

symmetrical ethyl methyl ethylene + Bromine → 2,3 di-brom pentane.

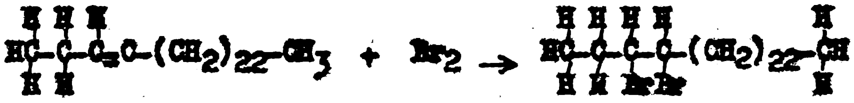
(B.P. 36°C)



$$HCH_2-CH=CH-(CH_2)_5-CH_3 + Br_2 \rightarrow HCH_2-CHBr-CHBr-CH_2-(CH_2)_5-CH_3$$

decylene (B.P. 172°C) + Bromine → 2,3 di-brom decane.

(1) Compounds with one double bond (Continued)

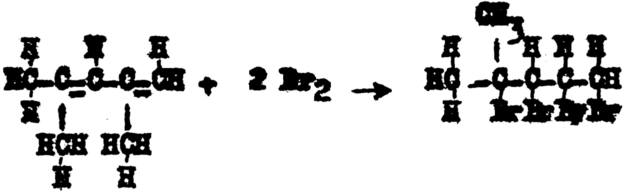


$$H_3C-CH_2-CH=CH-(CH_2)_{22}-CH_3 + Br_2 \rightarrow H_3C-CH_2-CHBr-CHBr-(CH_2)_{22}-CH_3$$

cerotene (a solid) (M.P. 58°C)

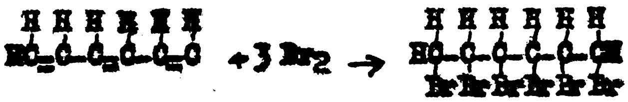
(2) Compounds with two double bonds such as

1,1,3 tri methyl buta-diene (B.P. 93°C)



$$H_3C-C(CH_3)=C(CH_3)-CH=CH + 2Br_2 \rightarrow H_3C-C(CH_3)Br-CBr-CHBr-CHBr$$

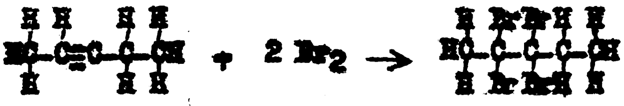
(3) Compounds with three double bonds.



$$HC=CH-CH=CH-CH=CH + 3Br_2 \rightarrow HCBr-CHBr-CHBr-CHBr-CHBr-CHBr$$

1,3,5 Hexatri-ene + Bromine → 1,2,3,4,5,6 brom hexane.

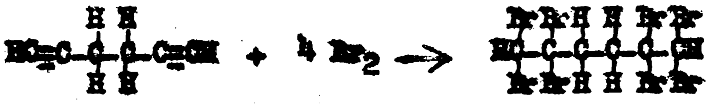
(4) Compounds with one triple bond (acetylene type).



$$H_3C-C\equiv C-CH_2-CH_3 + 2Br_2 \rightarrow H_3C-CBr_2-CBr_2-CH_2-CH_3$$

methyl ethyl ethylene

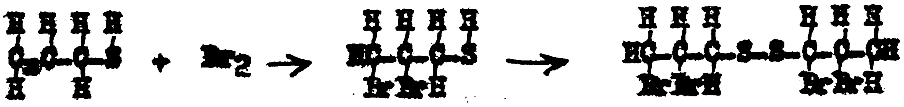
(5) Compounds with two triple bonds.



$$HC\equiv C-CH_2-CH_2-C\equiv CH + 4Br_2 \rightarrow HCBr_2-CBr_2-CH_2-CH_2-CBr_2-CHBr_2$$

1,5 hexa di-ine

(6) Action of Bromine on mercaptans sulfur derivatives.



$$H_2C=CH-CH_2-S + Br_2 \rightarrow H_2CBr-CHBr-CH_2-S \rightarrow H_2CBr-CHBr-CH_2-S-S-CH_2-CHBr-CH_2Br$$

From the above reactions it seems that the absorption of bromine by the asphaltic oils ought to be some indication of unsatured hydrocarbons present, which are considered to be the compounds that are more easily

weathered or broken down, and a means of determining which are the better oils to be used in road construction.

The following procedure was used in performing the bromine absorption test. About ½ gm of oil was weighed out accurately in a flask and 25 cc of carbon disulphide added to dissolve the oil. Then 60 cc of bromine solution (2 cc of bromine to 750 cc of carbon disulphide) was added to the flask. The flask was corked and shaken up well before it was placed in a dark cabinet for 15 minutes. At the end of the 15 minutes it was removed from the dark cabinet and an excess of potassium iodide (100 cc of 40 gms K.I. per 1000 cc of water) was added. The mixture was shaken up well and then titrated with sodium thiosulphate solution (decinormal - 24.8 gms of sodium thiosulphate to 1000 cc of distilled water) until the external starch indicator didn't turn a blue color but remained perfectly clear. The difference in the two titrations give the amount of sodium thiosulphate necessary to absorb the amount of bromine not absorbed by the sample of oil.

To find the amount of bromine absorbed by the oil, it was necessary to standardize the bromine solution by adding an excess of the potassium iodide solution to 60 cc of the bromine solution and titrating this mixture until the starch indicator showed a neutral condition. This would give the total amount of bromine available for absorption so the difference between the standardized value and the value for the oil absorption will give the number of cc's absorbed by that quantity of oil.

In order to get the bromine absorption value it was necessary to multiply the number of cubic centimeters absorbed by the factor (.008) to get the number of grams of bromine absorbed, and then to find the amount of bromine that would be absorbed by titrating 100 grams of the oil.

The amount absorbed by 100 grams of the oil is the bromine absorbed value of that oil. The bromine absorption values were determined for all the different oils in each different test and the results recorded.

The visual results of the absorption test were noted and the residues saved for further testing.

It may be well to note at this point that a great many of the samples showed a large amount of scum or foam. This foam may be due to the phenomena that when non-pure liquids are mixed,[17] resistance is offered to them merging together and foam forms due to the thin films of the materials in the solution being of different concentration. The solute then either concentrates in the surface or recedes from the surface leaving the non pure parts as foam. It is a known fact that pure liquids will mix without any foam or scum being formed. This foam formed during the bromine absorption test may be some indication of the unsaturated hydrocarbons present in the asphaltic oils.

— DATA & RESULTS —

Manufacturers of the different oils.

A. — Texas Oil Company called Indiana surfacing material SC-4, manufactured from Mid-Continent crudes by the cracking process. Lockport Illinois Refinery.

B. — Standard Oil Company of Indiana, Whiting, Ind. Manufactured from Mid-Continent crudes. Supposed to a cracked product.

C. — Skelly Oil Company, El Dorado, Kansas Refinery. Manufactured from Mixed Kansas crude, using a vacuum refined asphalt, cut back with gas oil.

D. — Lion Oil Company, El Dorado, Arkansas. Manufactured from Urbana crude. It is a topped crude product.

E. — Lion Oil Company, El Dorado, Arkansas. Urbana crude was used. It was a blend containing 30% cracked tar and 70% uncracked crude bottoms.

F. — Lion Oil Company, El Dorado, Arkansas. This is a blend consisting of 100°F melting point topped Smackover crude and non-adjacent gas oil from Smackover crude.

G. — Shell Petroleum Corporation from their Norco, Louisiana refinery. It is a steam refined Mexican crude.

H. — Shell Oil Petroleum Corporation from their East Chicago Refinery and it is a mixture of cracked residues from mid-Continent crudes.

I. — Texas Oil Company from their Cody, Wyoming refinery. It is a steam distilled, manufactured from Oregon crudes (Oregon Basin Crudes).

J. — Standard Oil of California from their Richmond refinery. This is a blend consisting of 80% 14° gravity residuum and 20% of 50 penetration steam refined asphalt, both produced from Calif. crudes.

K. — Socony - Vacuum Oil Company from their Augusta, Kansas refinery. It is a product resulting from the vacuum and steam distillations of mixed base Kansas Crudes.

L. — Socony - Vacuum Oil Company from their Augusta, Kansas refinery. This is a blend consisting of residues from the cracking process and the vacuum steam distillation of Kansas mixed base crudes.

M. — Socony - Vacuum Oil Company from their Casper, Wyoming refinery. This is a straight run product of Wyoming crudes.

N. — Socony - Vacuum Oil Company from their Casper, Wyoming refinery. This is a blend of asphaltic residues manufactured by their new synthetic process. A cracked product.

O. — Gulf Refinery Company from their Cincinnati, Ohio refinery. A cracked product of mixed Mid-Continent crudes.

P. — American Bitumuls Company from their Baltimore, Maryland refinery. It is a California crude shipped to the Baltimore refinery and is made into a topped product.

Computations for Film Thickness on Ottawa Sand

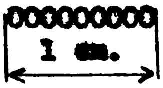
No. of Particles per gram = 1400

By check in 0.1 gm counting

$$\begin{array}{r} 142 \\ 138 \\ \underline{140} \\ 3\,\backslash\,\underline{420} \\ 140 \end{array} \qquad 1 \text{ gm} = 1400$$

Pass through 20 screen mesh 0.833 millimeter or .0328 inches close fit.

By calculation



12d = 10 mm.
d = 0.833 mm.

Assumption that many will be slightly less than 0.833 so average closer to 0.80 mm. = .0314"

Surface Area of one particle = $\pi d^2 = 3.1416\,(.0314)^2$

$= .003097$ sq. in.

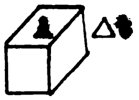
1400 Particles in a gram = 1400 (.003097)

∴ 1 gm. sand surface Area = 4.3358 sq. in. 1 gm.

$$\frac{500 \text{ gm. } (.433580 \text{ in}^2/\text{gm})}{144 \text{ in}^2/\text{sq.ft.}} = 15.05 \text{ sq. ft.}$$

500 gms. sand surface Area of 15.05 sq. ft. or 2,167.9 sq. in.

Film thickness if 3% oil added = 15.3872 cc. = 1 in³ approx.



$n(\Delta t) = 1$ inch

$\Delta t = \frac{1}{n}$

1 cu. in. = 1 in² $(n)(\Delta t)$

$n\Delta t = 1$ inch

$\Delta t = \frac{1}{2{,}167.9} = .0046''$

Heating in oven 1 gram of oils on tin plates until failure.

145°C.

Oil	Wt. oil gms.	Loss in Wt. in gms.	% loss of weight	Time to lose tackiness in hrs.	At end of 48 Hours in Oven
A	1.000	0.3264	32.64	15	Black, dusky, flakey surface
B	"	0.2422	24.22	37	Black shiny brittle cracked(sur-
C	"	0.2562	25.62	32	" " " " (face"
D	"	0.3580	35.80	34	" " " " "
E	"	0.2356	23.56	34	" " " " "
F	"	0.1896	18.96	34	" " " " "
G	"	0.3620	36.20	32	" " " " "
H	"	0.3467	35.67	24	Black, dusky, flakey surface
I	"	0.3388	33.88	40	Black shiny brittle cracked(sur-
J	"	0.3290	32.90	40	" " " " (face"
K	"	0.2933	29.33	32	" " " " "
L	"	0.3238	32.38	22	Gray, dusky, hard but no cracks
M	"	0.3953	39.53	40	Black, shiny, brittle cracked (sur-
N	"	0.3272	32.72	40	" " " " (face"
O	"	0.2624	26.24	20	Gray, dusky, soft, flakey surface
P	"	0.3304	33.04	32	Black, shiny, brittle cracked(sur- (face

After being in the oven 15 hours oils A, L, and O began to lose their tackiness but remained black and glossy on the surface and they were the first to fail as shown above.

Oils I, J, M & N at the end of 24 hours still retained their tackiness while all the other oils had failed or began to fail.

The final characteristics of all the oils at the end of the test which lasted 48 hours, is shown in the table above.

Heating in oven a 3% oil film on Ottawa Sand.

143°C.

Oil	Wt. oil gms.	Loss in Wt. in gms.	% loss of Wt.	Time of failing hrs.
A	.6000	.2192	36.5	45
B	.5500	.3464	63.0	130
C	.5500	.2009	36.4	74
D	.6300	.2900	46.2	124
E	.5400	.1706	31.5	93
F	.5000	.0562	1.2	69
G	.6300	.2286	36.3	55
H	.6200	.2789	45.0	32
I	.6000	.2176	36.3	46
J	.6000	.3255	54.7	90
K	.5600	.2754	49.3	51
L	.6000	.1404	23.4	32
M	.6700	.1938	29.0	56
N	.6000	.1591	26.5	38
O	.5500	.3358	61.1	103
P	.6000	.0110	1.8	96

Visual characteristics of mixtures during test.

After 12 hours heating oils - A. F. H, and L were losing tackiness and becoming hard and brittle. At end of 18 hours - oils A, F, H, and L were becoming quite hard while the remainder of oils began to show signs of losing their tackiness except B, D, and O.

Oil L at end of 32 hours had lost its adhesiveness between particles but it didn't become hard and brittle as did the other oils.

Oil O upon failure had turned very black and lost its adhesiveness but still remained oily instead of becoming hard and brittle.

Typical form of laboratory notes on Bromine Absorption Test.

Test on Original Oils

Oil	Wt. of oil in gms.	Burette Reading (1)	Burette Reading (2)	Difference (2) - (1)	Amt. Br. Absorbed in cc.
SS_1		0.0	33.8	33.8	
A	0.3078	8.3	29.2	20.9	12.9
B	0.3937	0.0	24.8	24.8	9.0
C	0.3244	0.0	26.7	26.7	7.1
D	0.3666	1.2	29.7	29.5	4.3
E	0.4288	0.0	25.2	25.2	8.6
F	0.4260	0.6	29.4	28.8	5.0
SS_2		0.0	10.3	10.3	
G	0.2986	3.2	7.7	4.5	5.8
H	0.4691	0.0	2.3	2.3	8.0
I	0.2668	43.7	47.9	4.2	6.1
J	0.4731	39.0	43.7	4.7	5.6
K	0.3800	34.2	38.8	4.6	5.7
L	0.3421	31.0	34.2	3.1	7.2
M	0.3956	27.5	30.9	3.4	6.9
N	0.4772	24.7	27.5	2.8	7.5
O	0.4994	22.0	24.7	2.7	7.6
P	0.6313	20.6	22.0	1.4	8.9

Test on oils from weather-ometer plates run 83 hours at 34°C.

Oil	Wt. of oil in gms.	Burette Reading (1)	Burette Reading (2)	Difference (2) - (1)	Amt. Br. Absorbed in cc.
SS_1		13.5	27.4	13.9	
A	0.0632	0.5	13.0	12.5	1.4
B	0.1946	13.2	24.4	11.2	2.7
C	0.0674	24.4	37.0	12.6	1.3
D	0.0486	0.0	12.3	12.3	1.6
E	0.1068	12.5	24.8	12.3	1.6
SS_2		24.8	40.4	15.6	
F	0.1354	0.1	13.8	13.7	1.9
G	0.1078	13.8	27.2	13.4	2.2
H	0.1020	27.2	39.5	12.3	3.3
I	0.0950	0.5	13.8	13.3	2.3
J	0.1160	14.2	27.5	13.3	2.3
K	0.0850	27.5	41.6	14.1	1.5
L	0.0778	0.2	13.7	13.5	2.1
M	0.1027	14.0	27.0	13.0	2.6
N	0.0732	27.1	40.7	13.6	2.0
O	0.1444	0.0	12.0	12.0	3.6
P	0.1721	12.2	23.9	11.7	3.9

Bromine Absorption Test on the original oils.

Oil (original)	Wt. of test sample	Part of 100 gms.	Absorbed gms. of Br.	Bromine Absorption Value
A	.1052	324.88	34.1774	0.3078
B	.0720	254.00	18.2880	0.3937
C	.0568	308.26	17.5092	0.3244
D	.0344	272.77	9.3833	0.3666
E	.0688	233.21	16.0448	0.4288
F	.0400	234.74	9.3896	0.4260
G	.0464	334.89	15.5389	0.2986
H	.0640	213.17	13.6429	0.4691
I	.0488	374.81	18.2907	0.2668
J	.0448	211.37	9.4694	0.4731
K	.0456	263.16	12.0000	0.3800
L	.0576	292.31	16.8370	0.3421
M	.0552	252.78	13.9535	0.3956
N	.0600	209.55	12.5730	0.4772
O	.0608	200.24	12.1746	0.6313
P	.0712	158.40	11.2781	0.4994

Bromine Absorption Test on Samples from continuous weathering under Arc Lamp for 83 hours at 34°C.

Oil	Wt. test sample in gms.	Equivalent Wt. of original oil	Part of 100 gms. of orig. oil	Absorbed gms. of Bromine	Bromine Absorption Value
A	.0746	.0632	1582.27	.0112	17.72
B	.1926	.1946	513.87	.0216	11.10
C	.0692	.0674	1483.67	.0104	15.43
D	.0486	.0486	2057.61	.0128	26.34
E	.1066	.1068	936.32	.0128	11.98
F	.1372	.1354	738.55	.0152	11.23
G	.1102	.1078	927.64	.0176	16.33
H	.1020	.1020	980.39	.0264	25.88
I	.1002	.0950	1052.63	.0184	19.37
J	.1212	.1160	862.06	.0184	15.86
K	.0870	.0850	1176.47	.0120	14.12
L	.0792	.0778	1285.34	.0168	21.59
M	.1047	.1027	973.71	.0208	20.25
N	.0758	.0732	1366.12	.0160	21.86
O	.1482	.1444	692.52	.0288	19.94
P	.1721	.1721	581.05	.0312	18.13

Visual characteristics of oils on plates under Arc Lamp.

After 4½ hours running oils B, D, and P began to become spotted on the plates. The final results according to looks may be classified as follows:

extra heavy spots: - B, D, and P.
" " : - F.
light " " : - E and J.
medium " : - A, I, L, and M.
light " : - C, G, H, K, N, and O.

The oils still were quite tacky when the plates were removed from under the Arc Lamp.

Bromine Absorption Test on samples from under carbon Arc Lamp Weather-Ometer Test for 630 hours at 140°C.

Oil	Wt. test sample in gms.	Part of 100 gms.	Amount Bromine Absorbed	Bromine Absorption Value
A	0.4336	230.63	.0344	7.93
B	0.3796	263.43	.0176	4.63
C	0.3486	286.86	.0440	12.62
D	0.3589	278.63	.0344	9.58
E	0.4744	210.79	.0344	7.25
F	0.2934	340.83	.0096	3.27
G	0.4434	225.52	.0526	11.86
H	0.2906	344.11	.0272	9.36
I	0.3077	324.99	.0280	9.10
J	0.2989	334.56	.0152	5.06
K	0.3258	306.93	.0152	4.67
L	0.1696	589.62	.0224	13.21
M	0.2004	499.00	.0160	7.98
N	0.1847	541.42	.0280	15.16
O	0.1834	545.25	.0232	12.65
P	0.3078	324.88	.0304	9.88

Bromine Absorption Test on samples of oils after failure in oven at 143°C.

Oil	Wt. test sample	Equiv. Wt. of original oil	Part of 100 gms. of original oil	gms. Bromine Absorbed	Bromine Absorption Value
A	.4376	0.6496	153.94	.0648	9.98
B	.3619	0.4776	209.38	.0464	9.72
C	.3272	0.4339	230.46	.0368	8.48
D	.3000	0.4672	214.04	.0240	5.14
E	.4398	0.5753	173.82	.0512	8.90
F	.3176	0.3919	255.16	.0232	5.92
G	.4054	0.6354	157.38	.0440	6.92
H	.4352	0.6661	150.12	.0496	7.45
I	.3153	0.4785	208.98	.0384	8.02
J	.3006	0.4479	223.26	.0264	5.89
K	.3753	0.5310	188.32	.0312	5.88
L	.4542	0.6716	148.89	.0520	7.74
M	.3892	0.6436	155.37	.0448	6.96
N	.2702	0.4016	249.00	.0328	8.17
O	.3824	0.5184	192.90	.0456	8.80
P	.3057	0.4565	219.05	.0472	10.34

Bromine Absorption Test on 3% oil film on Ottawa Sand in oven 143°C.

Oil	Wt. test sample	Equiv. Wt. of original oil	Part of 100 gms. of original oil	Absorbed gms. of Bromine	Bromine Absorption Value
A	.3808	.6000	166.67	.0544	9.07
B	.2036	.5500	181.81	.0632	11.49
C	.3491	.5500	181.81	.0336	6.01
D	.2400	.6300	158.73	.0256	4.06
E	.3694	.5400	185.18	.0368	6.81
F	.4438	.5000	200.00	.0448	8.96
G	.4014	.6300	158.73	.0472	7.49
H	.3411	.6200	161.29	.0432	6.97
I	.3824	.6000	166.67	.0592	9.87
J	.2715	.6000	166.67	.0456	7.60
K	.2846	.5600	178.57	.0328	5.86
L	.4596	.6000	166.67	.0328	5.47
M	.4762	.6700	149.25	.0600	8.95
N	.4407	.6000	166.67	.0552	9.20
O	.2142 gain	.5500	181.81	.0584	10.62
P	.5890	.6000	166.67	.0456	7.60

Bromine Absorption Test on road oils that had air blown through them at rate of 500 cc per min. for 2 hours at 180°C.

Oil	Wt. Test sample in gms.	Parts of 100 gms.	Amount of Bromine Absorbed	Bromine Absorption Value
A	0.2052	487.32	.0328	15.98
B	0.1676	596.65	.0240	14.32
C	0.1816	550.66	.0232	12.76
D	0.2585	386.84	.0200	7.73
E	0.2886	346.50	.0264	9.15
F	0.2000	500.00	.0184	9.20
G	0.2334	428.44	.0344	14.74
H	0.1464	683.06	.0312	21.31
I	0.1948	513.34	.0384	19.71
J	0.3107	321.85	.0416	13.39
K	0.3092	323.41	.0288	9.31
L	0.2900	344.82	.0320	11.03
M	0.3744	267.02	.0384	11.02
N	0.2939	340.25	.0360	12.24
O	0.2798	351.40	.0312	11.15
P	0.4689	213.26	.0432	9.21

Visual characteristics of results of Bromine Absorption Test on original oils.

Oil	Amt. of Black heavy liquid in residue	Scum that settles into layer above Black liquid.	Top layer of clear liquid
A	medium amount	very, very little amt. of brown scum.	Large amt. of very clear white liquid.
B	medium amount	very little amount of light scum.	" " "
C	medium large amount	little amount of very dense light brown scum	" " "
D	small amount of slightly brownish-black	very, very little light brown scum.	" " "
E	medium large amount	very little amount of light scum.	" " "
F	" " "	very little amount of light scum.	" " "
G	" " "	little amount of light dense scum.	medium amount of clear liquid.
H	" " "	medium amount of light brown scum.	medium small amount of clear liquid.
I	large amount	little amount of light dense scum.	medium amount of very clear liquid.
J	" "	very little amount of medium light scum.	" " "
K	" "	little amount of light very dense scum.	medium amount very clear liquid.
L	medium large amount	very little amount of light brown scum.	large amount of very clear liquid.
M	medium amount	very, very little light scum.	large amount of very clear liquid.
N	large amount	little amount of dense light scum.	medium amount of clear liquid.
O	medium amount	very large amount of dense light brown scum	very little liquid on top.
P	large amount	little amount of light porous scum.	medium amount of clear liquid.

Visual characteristics of results from Bromine Absorption Test on oils from carbon Arc Lamp that were weathered for 63 hours at 34°C continuous weathering.

Oil	Amount of heavy Brown liquid in bottom of flask	Amount of scum collected above heavy Black liquid	Amount of liquid contained in top layer
A	med. large amount reddish Brown liquid	very, very little light scum	large amount of very clear liquid on top
B	med. large amount Blackish Brown	" " " " " "	" " " " " " " "
C	med. large amount dark reddish Brown	" " " " " "	" " " " " " " "
D	med. large amount reddish Brown liquid	very little light porous scum	" " " " " " " "
E	medium amount of reddish Brown liquid	no scum	" " " " " " " "
F	Med. amount of dark reddish Brown liquid	no scum	" " " " " " " "
G	large amount reddish Brown liquid	very little light porous scum	medium amount of very clear liquid on top
H	medium large amount of reddish Brown	very little light dense scum	large amount of very clear liquid on top
I	medium amount of dark reddish Brown	no scum	" " " " " " " "
J	med. large amount dark reddish Brown	very, very little scum	" " " " " " " "
K	med. small amount reddish Brown liquid	" " " " " "	" " " " " " " "
L	med. small amount reddish Brown	" " " " " "	" " " " " " " "
M	large amt. reddish Brown liquid	little amount of light porous scum	medium amount of very clear liquid on top
N	med. small amount very reddish Brown liquid	very little amount of light scum	large amount of very clear liquid on top
O	large amount of very reddish Brown liquid	small amount of light porous scum	medium amount of very clear liquid on top
P	large amount of very reddish Brown liquid	small amount of light porous scum	" " " " " " " "

Visual characteristics of results from Benzine Absorption Test on oils from carbon Arc Lamp weather-ometer that was run 630 hours at 140 °F on modified weathering cycle.

Oil	Amount of heavy Black liquid in bottom of flask	Amount of scum collected above heavy Black liquid	Amount of liquid contained in top layer.
A	large amount	small amt. of very light porous scum	medium amt. of clear liquid on top
B	" "	large amt. of very light porous scum	very small amt. of clear liquid
C	" "	large amt. of med. light porous scum	" " " " " " " "
D	" "	large amt. of light Brown medium dense scum	" " " " " " " "
E	" "	med. amt. of very light porous scum	medium amt. of clear liquid
F	" "	med. amt. of very light med. dense scum	" " " " "
G	" "	large amt. of light Brown porous scum	small " " " "
H	medium "	medium amt. of light dense scum	medium " " " "
I	" "	small amt. of very light scum	large " " " "
J	" "	med. large amt. of very light porous scum	medium " " " "
K	med. large amt. of dark reddish Brown	very large amt. of very light dense scum	no liquid on top
L	med. large amt. of dark reddish Brown	small amt. of light very dense scum	large amt. of clear liquid
M	very large amt. of Black liquid	med. large amt. of light medium porous scum	small " " " "
N	very large amt. of Blackish Brown liquid	med. large amt. of light very porous scum	" " " " "
O	very large amt. of Black liquid	med. large amt. of extra light fairly porous scum	" " " " "
P	small amt. of Black liquid	very large amt. of light Brown dense scum	no liquid on top

Visual characteristics of Bromine Absorption Test on oils from films tested in oven until failure.

Oil	Amount of heavy Black liquid in bottom of flask	Amount of scum collected above heavy liquid	Amount of liquid contained in top layer
A	large amt. of Black liquid	large amt. of med. dark dense scum	very little liquid on top
B	" " " " "	small amt. of light Brown porous scum	large amt. of clear liquid on top
C	" " " " "	med. amt. of light porous scum	medium amt. of clear liquid on top
D	small amt. of Black liquid	small amt. of light dense scum	large amt. of clear liquid on top
E	med. amount of Black liquid	large amt. of light porous scum	small amt. of clear liquid on top
F	med. amount of Black liquid	large amt. of light Brown dense scum	small amt. of clear liquid on top
G	med. amt. of Black liquid	large amt. of light porous scum	no liquid on top
H	med. small amt. of Black liquid	med. amt. of Brown dense scum	med. amt. of clear liquid on top
I	large amt. of Black liquid	med. large amt. of light porous scum	small amt. of clear liquid on the top
J	med. small amt. of Black liquid	large amt. med. light dense scum	very little liquid on the top
K	large amt. of Black liquid	med. amt. of light colored fairly dense scum	med. amt. of very clear liquid above
L	med. small amt. of Black liquid	large amt. of Brown, very dense scum	very little clear liquid on the top
M	large amt. of reddish Brown liquid	large amt. of medium light, porous scum	small amt. of clear liquid on top
N	large amt. of Black liquid	large amt. of medium light, porous scum	small amt. of clear liquid on top
O	large amt. of Brown solid	large amt. of Brown flaky dense scum	small amt. of clear liquid on top
P	large amt. light Brown liquid	very small amt. of very light scum	large amt. of clear liquid on top

Visual characteristics of results from Bromine Absorption Test on oils from test with Ottawa Sand.

Oil	Amount of heavy Black liquid in bottom of flask	Amount of scum collected above heavy Black liquid	Amount of liquid contained in the top layer
A	very little	very large amt. of dark brown dense scum	no liquid
B	large amount	med. large amt. of light Brown dense scum	very, very little liquid on top
C	" "	little amt. of light porous scum	little clear liquid on top
D	small "	very large amt. of med. dark Brown dense scum	no liquid on top
E	" "	med. large amt. light Brown dense scum	" " " "
F	med. small amount	med. large amt. light Brown dense scum	" " " "
G	med. amt. of reddish Brown liquid	small amt. of Black dense scum	med. amount of very clear liquid
H	small amount	med. large amt. Brown dense scum	no liquid on top
I	medium "	med. large amt. of light Brown porous scum	" " " "
J	small "	large amt. of light med. porous scum	" " " "
K	medium "	med. small amt. light porous scum	small amt. of clear liquid on top
L	" "	med. amt. of very dense Brown scum	a little liquid on top
M	large "	large amt. of light Brown med. porous scum	very, very little liquid on top
N	medium "	med. amt. of light Brown dense scum	no liquid on top
O	large "	very large amt. very dense Brownish scum	" " " "
P	medium "	very large amt. med. dense light Brown scum	" " " "

Visual characteristics of Bromine Absorption Test on oils that have had air blown through them for 2 hours at rate of 500 cc per min at 180°C.

Oil	Amount of heavy black liquid in bottom of flask	Amount of scum collected above black liquid	Amount & kind of liquid in top layer
A	large amount	med. amt. of light brown porous scum	small amt. of clear liquid on top
B	" "	very little amt. of light scum	large amt. of clear liquid on top
C	" "	small amt. of very light dense scum	" " " " " " "
D	" "	very, very little light scum	" " " " " " "
E	medium "	very, very little light porous scum	" " " " " " "
F	large "	very little light porous scum	" " " " " " "
G	very large amount	med. small amt. of light brown dense scum	med. large amt. of clear liquid on top
H	large amount	med. amt. of light brown dense scum	med. small amt. of clear liquid on top
I	med. large amount	very, very little light colored dense scum	large amt. of very clear liquid
J	large amount	med. large amt. of light porous scum	med. small amt. very clear liquid on top
K	" "	small amt. of light porous scum	med. large amt. of clear liquid on top
L	medium "	med. amt. of light dense scum	med. amt. of very clear liquid on top
M	large "	small amt. of light very porous scum	med. small amt. of clear liquid on top
N	medium "	small amt. of light dense scum	med. small amt. of clear liquid on top
O	small "	med. amt. of light med. dense scum	small amt. of clear liquid on top
P	" "	very large amt. of light very porous scum	very small amt. of liquid on top

Table of laboratory characteristics of oils and test results.

Process	Steam & Vacuum Distillation					
Oil	D	G	I	K	M	P
Specific Gravity 25°/25°C.	0.989	0.984	0.990	1.009	0.996	0.976
Water & sediment, per cent	0.05	0.05	0.05	0.2	0.05	0.0
Flash point, (Cleveland open cup). °C	214	140	140	150	174	205
Viscosity, Saybolt Furol, 60°C, sec.	420	593	498	435	663	532
Heterogeneity Test	neg.	neg.	neg.	neg.	neg.	neg.
Distillation 0° to 225°C %	0.0	0.0	0.0	0.0	0.0	0.0
0° to 315°C %	0.0	3.0	trace	trace	0.0	0.0
0° to 360°C %	0.5	12.0	2.5	1.2	trace	0.5
Test on Distillation Residue Bitumen Soluble in CCl_4,%	99.94	99.92	99.93	99.95	99.93	
Tests on 100 Penetration Res. Actual penetration of Res.	90	107	99	96	98	
Heat time to above Res. hrs.	22	3	9	4.75	6	
Total Residue, %	75.5	75.3	76.2	77.4	76.3	
Ductility, 25°C, 5cm/min, cms	146	125	106	150	94	
" , 4°C, 5cm/min, cms	7.0	9.5	9.5	7.0	9.0	
Asphaltenes, per cent	6.46	20.42	13.14	11.78	14.07	5.08
Bromine Absorption Test - gms. per 100 gms. original oil	9.38	15.54	18.29	12.00	13.95	11.28
oven test, 143°C	5.14	6.92	8.02	5.88	6.96	10.34
Ottawa Sand test, 143°C	4.06	7.49	9.87	5.86	8.95	7.6
partial failure under Arc Lamp	26.34	16.33	19.37	14.12	20.25	17
complete failure under Arc Lamp	9.58	11.86	9.10	4.67	7.98	
air blown - 500 cc per min for 2 hrs at 180°C	7.73	14.74	19.71	9.31	11	

Table of laboratory characteristics of oils and test results, (cont'd.)

Process	Blend steam refined Residues	Cut back steam Residue & gas oil		Blend steam refined cracked Residues		
Oil	J	F	G	L	B	E
Specific grav. 25°/25°C	0.972	0.976	0.979	1.025	0.992	0.995
Water & sediment, percent	0.0	0.0	0.4	0.0	0.3	0.0
Flash point, (Cleveland open cup) °C.	156	159	148	154	130	145
Viscosity Saybolt Furol, 60°C, sec.	522	552	465	472	446	649
Heterogeneity Test	neg.	neg.	neg.	pos.	slightly pos.	slightly pos.
Distillation 0° to 225°C %	0.0	0.0	0.0	0.0	0.0	0.0
0° to 315°C %	0.0	0.5	0.2	trace	1.5	0.0
0° to 360°C %	1.5	3.0	7.0	1.0	7.0	0.5
Test on Distillation Res. Bitumen sol. in CCl_4 %	99.94	99.96	99.93	99.99	99.93	99.97
Tests on 100 Penetration Residue. Actual penetn. of res.	93	107	93	100	105	93
Heat time to above pen., hrs.	7	9.5	6	3	11	11.5
Total Residue, %	79.7	82.3	78.8	80.1	79.1	80.1
Ductility, 25°C, 5cm/min, cms	95	140	111.0	140	200	110
" , 4°C, 5cm/min, cms	7.0	9.5	5.5	7	5.5	6.5
Asphaltenes, per cent	10.00	8.61	12.66	14.66	8.95	8.80
Bromine Absorption Test- gms. per 100 gms. original oil	9.47	9.39	17.91	16.84	18.29	16.04
oven test, 143°C	5.89	5.92	8.48	7.74	9.72	8.90
Ottawa Sand test, 143°C	7.60	8.96	6.01	5.47	11.49	6.81
partial failure under Arc Lamp	15.86	11.23	15.43	21.99	11.10	11.98
Complete failure under Arc Lamp	5.06	3.27	12.62	13.21	4.63	7.25
air blown - 500 cc/min for 2 hrs at 180°C	13.39	9.20	12.76	11.03	14.32	9.15

Table of laboratory characteristics of oils and test results, (cont'd.)

Process	Blended cracked Residues		Straight cracked	
Oil	N	M	A	O
Specific gravity, 25°/25°C	0.999	1.052	1.082	1.031
Water & sediment, percent	0.0	0.02	0.0	0.4
Flash point (Cleveland open cup), °C.	170	155	152	210
Viscosity Saybolt Furol, 60°C, sec.	637	467	480	[illegible]
Heterogeneity Test	pos.	pos.	pos.	pos.
Distillation 0° to 225°C %	0.0	0.0	0.0	0.0
0° to 315°C %	trace	trace	0.0	0.0
0° to 360°C %	0.5	3.5	1.8	0.5
Test on Distillation Residue Bitumen soluble in cc/4, %	99.94	99.85	99.96	99.52
Tests on 100 Penetration Residue Actual penetration of residue	91	94	95	107
Heat time to above pen., hrs.	4.5	2.5	1.5	7
Total Residue, percent	81.2	81.8	84.0	84.6
Ductility, 25°C, 5cm/min-cms	150	133	105	70
" , 4°C, 5cm/min-cms	5.5	1.0	0.0	4.5
Asphaltenes, percent	13.04	19.05	26.00	13.29
Bromine Absorption Test - gms. per 100 gms. original oil	12.57	13.64	34.18	12.17
oven test, 143°C	8.17	7.45	9.98	8.80
Ottawa Sand test, 143°C	9.20	6.97	9.07	10.62
partial failure under Arc Lamp	21.86	25.88	17.72	19.94
complete failure under " "	15.16	9.36	7.93	12.65
air blown - 500 cc/min for 2 hrs. at 180°C.	12.24	21.31	15.98	11.15

CONCLUSIONS

Heating in the Oven.

1. The ultimate loss from exposure in the oven was somewhat greater than the total loss on exposure in the distillation residue test.

2. The residues from film exposure were much harder than the residues from the distillation test, this has been shown to be due to the increased free carbon content from oxidation, carbonization and polymerization.

3. The percent loss in weight from films 0.025 inch thick and films 0.0005 inch thick on Ottawa sand was very nearly the same for every oil except oils B, J and O which had an increased loss from 90 to 100 percent. The reason for oils J and O not becoming hard and brittle upon failure may be due to the viscosity of the asphaltenes in them not becoming any greater after reaching a definite consistency.

4. Thin films retain their life and adhesiveness longer than heavier films as was shown by the time it took the films on the Ottawa sand to fail in comparison to the plain film in the pans.

5. The chemical activity or bromine absorption actually decreased approximately 50 percent from the original oils for all thickness of films tested. This shows that the samples are much more saturated and have become highly polymerized, which usually occurs at high temperatures.

The Weather-Ometer.

1. The oils that gained the most weight were found to be the most chemically active, while oils that remained constant in weight showed no marked change.

2. Thin films seemed to retain their adhesiveness and tackiness better than the thicker films.

3. In the case where the films were thin the chemical activity actually

increased considerably, whereas with the thicker films that were weathered for a much longer period of time showed very little change from the original oils.

4. The gain in weight of the films is probably due to the ozonides formed when ozone (liberated from the arc) reacted with the unsaturated constituents of the oils. This was ascertained by treating the weathered oils with water to break up the ozonides forming water and soluble aldehydes which precipitate free silver in the presence of ammonical silver nitrate.

This was found not to be true in the case of oils that did not gain in weight.

In General

1. It seems that the oils under any test have to lose a certain amount of volatile matter before losing their adhesiveness.

2. Since bromine absorption is a test of the unsaturatedness of a hydrocarbon more should be done with it to ascertain its value as determining the quality of a good oil.

3. The original oils bromine absorption values seem to fall in a trend similar to the percent of asphaltenes in the oils. This suggests that the asphaltenes may contain the unsaturated carbon atoms of the bituminous material.

4. The general indication is that under the action of the weather-ometer oxidation takes place while in case of the oven the changes lead to polymerization and carbonization.

5. The chemical activity of the oils after having air blown through them remained approximately the same as the original oils which indicates that although oxidation took place polymerization took place to a sufficient extent so that the saturation of the oils remained the same at the end of the test as at the beginning.

6. After examining all the laboratory tests and results of experimental data, the author suggests that more research work be done to develop a good laboratory test so that the differences in the various asphaltic materials may be recognized from the residues obtained from the laboratory test.

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