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**ACCELERATED WEATHERING PROPERTIES**  
**Of**  
**OKLAHOMA ASPHALTS**

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# ACCELERATED WEATHERING PROPERTIES OF OKLAHOMA ASPHALTS

## INTRODUCTION

### ACKNOWLEDGMENTS

Grateful acknowledgment is due to Mr. Fred W. Padgett and to Dr. C. T. Langford for the supervision of the work done on the Oklahoma asphalts. Further thanks are due Dr. Charles N. Gould of the Oklahoma Geological Survey for his interest and financial aid through the Survey toward the collection of samples for this problem. Much credit is also due H. N. Peters, Jr. for analytical work on the samples and Mr. Ray L. Six for taking photographs.

The laboratory work was done under a Graduate Research Fellowship of the University of Oklahoma. The problem was conceived and suggested by Prof. Padgett and the work outlined by him.

Acknowledgement is also made for the cooperation extended by the School of Petroleum Engineering of the University, in the course of this work.

### PURPOSE

Herbert Abraham<sup>1</sup> says, in connection with a discussion of the slight effect caused by air blowing of extracted natural asphalts from Oklahoma; "This is further corroborated by the author's observations on paints made from the extracted sand asphalts which were found to be *highly resistant* to atmospheric oxidation." The object of this research is to develop and apply an accelerated weathering test to these products and to attempt to either prove or disprove this statement, by bringing about a comparison of their weathering properties with those of petroleum and some other naturally occurring asphalts. No attempt is made to delve deeply into the theoretical considerations coincident with weathering in general, nor has any differentiation been made between the effects of the different weathering agents.

There are, in southern Oklahoma, vast deposits of both sand and rock asphalt varying in bitumen content from a trace up to about fifteen per cent, averaging possibly six or seven per cent. The extent of these deposits has been estimated variously from one to thirteen million tons.<sup>2</sup>

At present very little natural asphalt is being used. There are three quarries in operation, two at Dougherty in Murray County

## FOREWORD

This report on the weathering properties of Oklahoma asphalts represents a survey undertaken in the fall of 1928. The work is being continued to clear up certain points and to include other natural bitumens of Oklahoma.

Before the conclusions can be accepted as final, several points will have to be explained and the scope of the tests broadened. The results probably represents a fair comparison of weathering properties where the material is used on steel exposed to the air, but not necessarily for impregnated roofing materials or for pipe covering where the pipe is buried. Why the weathering progresses so rapidly just after water application, also must be explained.

Fred W. Padgett

Norman, Oklahoma  
October, 1929.

<sup>1</sup> Abraham, Herbert, Asphalts and allied substances: D. Van Nostrand Co., p. 100, 1920.

<sup>2</sup> Snider, L. C., Rock asphalts of Oklahoma and their use in paving: Oklahoma Geol. Survey, Circ. 5, 1914.

and one northwest of Ardmore in Carter County. This material is being used for paving. However, there is a vast field for the use of asphalt other than paving. If it can be established that natural rock or sand asphalts from Oklahoma are good from a standpoint of weather resisting qualities (this is, sufficiently good to warrant their use at an increased production cost), it may mean the development of another great industry in Oklahoma.

The field for the use of asphalts includes the manufacture of bitumenized roofing material and all types of asphalt paints and protective coatings for the painting of steel, ship hulls, etc. All of these services require the greatest available weather resisting qualities. The weather resisting qualities desirable in paving are obvious.

### PREVIOUS WORK

Up to this time no reports have been found of work done on Oklahoma asphalts to determine their weathering characteristics other than that referred to in which Abraham<sup>3</sup> tried to raise the softening point and increase the hardness of some extracted asphalt from near Ardmore by blowing air through it in the same manner as in the manufacture of petroleum asphalts. This attempt was unsuccessful, showing the asphalt to be difficult to oxidize.

There has been some work done on the effect of exposure on bitumens.<sup>4</sup> This was carried out by placing the samples on watch glasses, in a dust-proof glass case, and exposing them to the sun for several months. The samples were then examined by observation and the gain in organic matter insoluble in CS<sub>2</sub> was noted. The authors described the sample of fluxed Bermudez with which they were working as "Portion glossy and dark brown and a patch rough." Later<sup>5</sup> the same type of test was run on more fluid bitumens. Further work was done using this same exposure chamber of tar products and the following conclusions were reached.<sup>6</sup>

1. Upon exposure to service conditions tar products materially harden to a much greater extent than can be attributed to loss of bitumen alone.
2. The changes which take place other than loss of distillate are accompanied by formation of organic matter insoluble in carbon disulfide.

More recently accelerated weathering has come into prominence in connection with paint manufacture. Nelson and Schmutz<sup>7</sup> have

<sup>3</sup> Op. cit., p. 100.

<sup>4</sup> Hubbard, Prevost and Reeve, C. S., Effect of exposure on bitumens: Jour. Ind. & Eng. Chem. vol. 5, p. 215, 1913.

<sup>5</sup> Reeve, C. S., and Lewis, R. H., Effect of exposure on some fluid bitumens: Jour. Ind. & Eng. Chem. vol. 9, p. 743, 1917.

<sup>6</sup> Reeve, C. S., and Anderton, B. A., Effect of exposure on tar products: Jour. Franklin Inst. Oct., 1916.

<sup>7</sup> Nelson, H. A., and Schmutz, F. C., Accelerated weathering: Jour. Ind. and Eng. Chem. vol. 18, p. 1222, 1926.

published a paper on accelerated weathering in which they emphasize the necessity for adjustment of the cycle to approximate outdoor conditions. In paint weathering it is necessary to enrich the air with oxygen to get results more nearly corresponding to actual outdoor tests.

Still more recently the Bureau of Standards has done some work on accelerated weathering of paints, varnishes, and bituminous materials which is published under the title "Accelerated Tests of Organic Protective Coatings."<sup>8</sup> In this paper an apparatus and method of procedure is described consisting of a drum or sample chamber where the materials are treated with light resembling sunlight, water spray, various gases, etc.

The apparatus used in this experiment is of a nature similar to that mentioned above. In correspondence from O. G. Strieter of the Bureau of Standards concerning this method of testing appears the following sentence; "The method for asphalts is essentially the same as for paints, only that ozone is not used and that a visual examination of the panel is sufficient for determining the durability of the asphalt." These assumptions have been accepted.

### PRELIMINARY WORK

#### COLLECTION AND DESCRIPTION OF SAMPLES

Two months were spent in the field during the summer of 1928 visiting every known location<sup>9</sup> of asphalt and asphaltite in Oklahoma, and sampling them where possible. Approximately 90 deposits were visited and 100 samples obtained. Some of the deposits are obviously valueless, but a large majority of them are of commercial proportions if a market could be provided. The asphalt area is shown on the accompanying map. It extends from the Arkansas line west to the vicinity of Granite in Greer County, and from Red River north almost to the thirty-fifth parallel, or about one-third of the north and south extent of the state. The deposits are located on a map (fig. 1) and those on which weathering tests were run are especially designated with a letter corresponding to their laboratory designation, described later.

In many instances it was impossible to get a very representative sample in the short time available and with the method employed. However, in the case of all the samples used in the tests particular care was exercised to obtain samples which were representative of their respective deposits. In choosing these samples, of which there are ten, the samples were, as far as possible, chosen

<sup>8</sup> Walker, Percy H., and Hickson, E. F., Accelerated tests of organic protective coatings: Dept. of Commerce, Bur. Standards, Res. Paper no. 1, 1928.

<sup>9</sup> Hutchinson, L. L., Preliminary report on the rock asphalt, asphaltite, petroleum, and natural gas in Oklahoma: Oklahoma Geol. Survey Bull. 2, 1914.

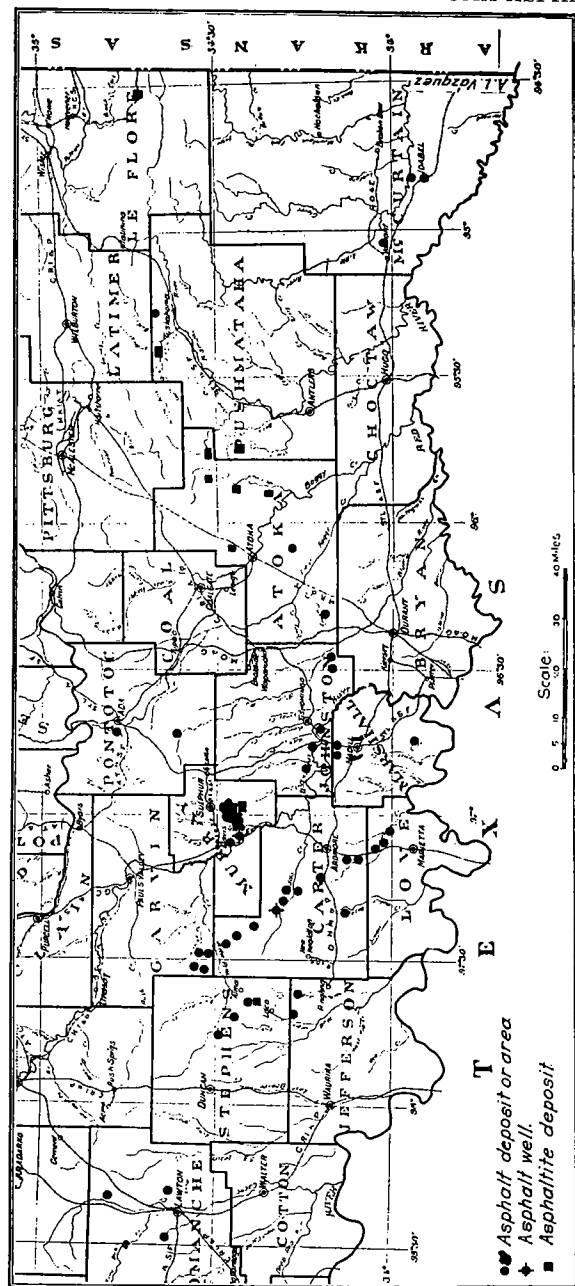


Figure 1.—Map of southeastern Oklahoma showing location of asphalt deposits.

which were representative of the deposits in a given district. Furthermore, all of these ten samples are from deposits which have commercial possibilities.

A short description of the occurrence of each of the samples chosen for weathering tests follows.<sup>10</sup>

#### SAMPLE A

LOCATION: SE  $\frac{1}{4}$  NE  $\frac{1}{4}$  sec. 18, T. 5 S., R. 1 W.

GEOLOGIC FORMATION: Clear Fork-Wichita formation, Permian age.

DESCRIPTION: This occurrence is about fourteen miles southwest of Ardmore. This asphalt is in a horizontal bedded sandstone saturated with a fairly soft bright black sticky bitumen. The deposit was worked about six years ago and a broad well-drained quarry remains. It is evident that a large amount of the material remains in place. The percentage bitumen in the sand is 13.35 per cent. A representative sample of this material was obtained.

#### SAMPLE C

LOCATION: NW  $\frac{1}{4}$  sec. 36, T. 1 N., R. 3 W.

GEOLOGIC FORMATION: Enid (Garber?) formation, Permian age.

DESCRIPTION: This occurrence is three or four miles southeast of Pernell. The asphalt is in a fine-grained sandstone, horizontally bedded, and varying in thickness from five to twenty-five feet. There are two small prospect pits, but no work has ever been done on a commercial scale. The deposit is located at the head of a ravine, and a quarry could be drained by gravity. There is evidence of the presence of a large amount of the material. The sample was taken from the outcrop and although the character of the bitumen contained is representative of the deposit, it is not certain that the sample obtained correctly represents the entire deposit as to saturation of the sand. The analysis showed 12.31 per cent bitumen.

#### SAMPLE D

LOCATION: SW  $\frac{1}{4}$  NE  $\frac{1}{4}$  sec. 36, T. 4 N., R. 5 E.

GEOLOGIC FORMATION: Ada formation, Pennsylvanian age.

DESCRIPTION: This occurrence is one or two miles west of Ada in Pontotoc County, and is representative of the occurrences in the

<sup>10</sup> Four other samples were originally chosen which more fully represented the entire asphalt-bearing territory, but three of these proved of almost liquid consistency and it was impossible to make a suitable test surface of them. The other sample, one from a pit operating northwest of Ardmore, was destroyed through accident in the extracting process.

Ada area. It is in a horizontally bedded sand and lime mixture, carrying by analysis 6.47 per cent bitumen. The thickness of the sand is unknown, but it exceeds twenty feet. The quarry from which this sample came has been operated by Ross Tipton of Ada and the material used without treatment for paving. Some very satisfactory pavements have been made from it. The bitumen content of the rock increases with depth according to the operator.

**SAMPLE E**

LOCATION: SW.¼ NE.¼ sec. 14, T. 1 S., R. 3 E.

GEOLOGIC FORMATION: Simpson formation, Ordovician age.

DESCRIPTION: This sample is from a prospect pit in a horizontally bedded sand and lime mixture occurring in the large asphalt district a few miles south of Sulphur in Murray County. The deposit appears to extend over several acres and shows an average thickness of something over ten feet. At first glance it would appear to be excellent for paving because of the large amount of limestone in the sand. The bitumen content is 10.74 per cent.

**SAMPLE F**

LOCATION: S.½ SW.¼ NE.¼ sec. 22, T. 1 S., R. 3 E.

GEOLOGIC FORMATION: Hunton (?) formation, Siluro-Devonian age.

DESCRIPTION: This sample is from the only deposit which has ever been developed in Oklahoma by underground mining methods. It is from the underground mine of the Gilsonite Paving and Roofing Company about five miles south of Sulphur which started operations about 1896. The mine has long since been abandoned. The bitumen occurs in a fine-grained, crystalline limestone. Tunnels follow the dip (20°) of the formation and the mine is mostly filled with water. It can be explored back to one working face about thirty feet from the entrance. The sample was taken from this face. The bitumen content is 5.83 per cent.

**SAMPLE G**

LOCATION: NW.¼ SE.¼ NW.¼ sec. 22, T. 1 S., R. 3 E.

GEOLOGIC FORMATION: Simpson formation, Ordovician age.

DESCRIPTION: This sample is from what is known as the Boline quarry in the asphalt district south of Sulphur, and is representative of the sand asphalt deposits in the Arbuckle Mountain region. The sand is made up of fine, rounded quartz grains, held together mainly by the bituminous material. The sand body dips about 25° SE. The thickness of the impregnated zone is over

twenty-five feet and the deposit is extensive. There is a good face exposed from which a representative sample could be obtained. The percentage bitumen in the sand is 9.37.

**SAMPLE H**

LOCATION: NE.¼ NW.¼ sec. 29, T. 1 S., R. 3 E.

GEOLOGIC FORMATION: Simpson formation, Ordovician age.

DESCRIPTION: This sample is from the Western Paving Company's "Sand Mine" in the Arbuckle Mountain district about three miles northeast of Dougherty. The sand member is similar in character to the previous deposit. This mine is now being worked as an open quarry. The face is about 30 by 100 feet and there is a 20- to 30-foot overburden of shale and conglomerate. The bitumen content varies in different parts of the face. A sample as nearly average as possible was taken. The bitumen content of this sample is 9.57 per cent.

**SAMPLE I**

LOCATION: SE.¼ sec. 25, T. 1 S., R. 2 E.

GEOLOGIC FORMATION: Viola limestone, Ordovician age.

DESCRIPTION: This sample is from the Western Paving Company's "rock mine" in the Arbuckle Mountain district about one mile northeast of Dougherty. This quarry is now in operation. There is a 90-foot face of limestone which is being quarried. The quarry is gravity drained. The material is crushed and mixed with sand asphalt and used for paving. The bitumen content of the limestone is 4.25 per cent.

**SAMPLE L**

LOCATION: SW.¼ sec. 27, T. 4 S., R. 8 E.

GEOLOGIC FORMATION: Trinity sandstone, Lower Cretaceous age.

DESCRIPTION: This deposit is of doubtful extent. It occurs about twelve miles east and four miles south of Tishomingo in Johnston County, and is representative of this district. The bitumen lies practically horizontal. It has been prospected slightly and found to be over a foot thick. Indications are that the deposit is fairly extensive from the fact that there is another deposit of similar character two miles west. The bitumen is the chief cementing material for the sand. Bitumen content of the sample obtained is 9.32 per cent.

## SAMPLE N

LOCATION: One-half mile east of Valliant, McCurtain County.

GEOLOGIC FORMATION: Trinity sandstone, Lower Cretaceous age.

DESCRIPTION: This appears to be an extensive deposit, and was mined several years ago by the open quarry method. Operations were suspended on account of difficulties in crushing the material. The occurrence is in a horizontal calcareous sand the thickness of which has not been determined. It is very thick. The bitumen content of the sample obtained is 12.35 per cent.

Samples which proved too liquid to run tests on were from the Trinity formation of southern Love County, from a quarry fourteen miles north of Lawton in Comanche County, from Maytubby Springs in Atoka County, and from a very rich deposit in the Trinity formation about three miles south of Tishomingo in Johnston County.

## EXTRACTION OF BITUMEN

The extraction of the bitumen from the samples was a long and tedious process, taking more time than any other single phase of the problem.

Two methods were tried: 1. Extraction with water, or more exactly, with a one per cent solution of sodium carbonate; and 2. Extraction with a solvent followed by distillation. Both methods were preceded by grinding or disintegrating the samples into as fine particles as possible. This, in most cases, was very difficult, due to the stickiness of the material. In carrying out the water extractions about ten pounds of the material was placed in the bottom of a three-gallon bucket and about two gallons of one per cent solution of sodium carbonate added. The mixture was put over a gas flame and heated to a temperature slightly below the boiling point of the solution and stirred. If the separation is successful the asphaltic material will come to the top and the sand will remain on the bottom of the bucket after a period of about thirty minutes. In most cases only a slight oily film formed on top of the water or else far too much sand remained in the mixture floating on the surface. These extractions were successful only in the case of unconsolidated sands, held together only by the bituminous material therein. Water extraction was notably successful in the case of the occurrences in the Trinity sand, both from Johnston and Love counties. It was partially successful in the case of the sand asphalts from the Simpson formation in the Arbuckle Mountain region. The separated material would have required

further treatment to remove excess sand, and this method was discarded for the separation of bitumen to be used for the weathering tests. Water extraction does not work on consolidated sands or relatively hard asphalts.

All of the material used in the weathering tests was extracted with either benzene or carbon tetrachloride. The samples were ground and mixed with the solvent and then run through a centrifugal filter which separated the sand. No attempt was made to clean the sand, but possibly 90 per cent of the bitumen was dissolved out of it. The solution or filtrate was collected and the solvent distilled off in a 500 cc. distilling flask connected to an ordinary glass water-cooled condenser. At first much difficulty was experienced in removing the last traces of solvent from the bitumen. When the distillation had proceeded to the point where the temperature began to rise markedly above the boiling point of the pure solvent the material in the flask tended to become frothy and foaming over was a common occurrence. This was finally overcome by running a stream of gas through the asphalt during the latter part of the distillation which aided in carrying over the last traces of solvent. The maximum temperature attained was 350° F. which is well below the temperature of cracking of the asphalt. This temperature was held approximately thirty minutes with a stream of gas bubbling through the material and in most cases this treatment removed any tendency to froth and the asphalt settled down to a smooth, consistent liquid containing no bubbles. Sample A was a notable exception to this and it was necessary to hold it in an oven at 120° F. for about twelve hours before the last traces of solvent were removed. This accounts for the fact that Sample A shows a hardness in the analysis which is doubtless greater than its natural hardness, the light fractions being distilled off in the extraction process.

A number of the samples as extracted proved too soft to form a coating on the sample plates which would not melt and run under the effect of the heat from the light employed in weathering, and these were reduced by vacuum distillation until they were sufficiently hard. It was necessary to reduce samples D, E, F, G, H, I, and L, to a consistency of 20 or greater in this manner.

The petroleum asphalts used in the weathering tests were made from California crude oils and are characterized by low sulfur content, high ductility, and low melting point for a given penetration.

Sample "Z" is a steam refined asphalt of penetration 2, and is a material furnished to paint and varnish manufacturers.

Sample P<sub>1</sub> has a penetration of 11. It is an air refined or blown asphalt and is used for roofing and similar work where a high melting point and properties of elasticity are necessary.

Sample P<sub>2</sub> has a penetration of 15 and is a steam refined, relatively hard asphalt also used in paint and varnish manufacture.

Sample P<sub>3</sub> has a penetration of 27. It is used for roofing.

Samples P<sub>4</sub> and P<sub>5</sub> have penetrations of 56 and 141, and are used for paving. The P<sub>4</sub> grade is used in asphaltic concrete construction and the P<sub>5</sub> grade is used in asphalt macadam work.

The other asphalts used in the comparative tests were refined native asphalts from foreign sources having penetrations of 3 and 15 respectively.

## LABORATORY WORK

### APPARATUS

The apparatus was designed with the idea of treating the samples with light and the attendant heat and with water, these being considered the most important weathering agents. The container consists of a metal can 24 inches high by 20 inches in diameter, with a cover through which a twelve-inch hole is cut allowing the light source to pass through. Brackets are soldered in the can ten inches from the bottom, and a perforated disk of the same diameter for the support. This disk serves as a shelf or false bottom on which the samples are placed. The perforation allows the circulation of air and the passage of water through the shelf into the bottom compartment during the water treatment. Just below the sample shelf a number of one-half-inch holes for the purpose of ventilation are drilled into the can. There is also a larger hole cut near the bottom of the can for the escape of the water.

The light source used was a flaming carbon arc without the globe. The carbons used were photographic white flame cored carbons,  $\frac{3}{8}$ -inch in diameter. This type of arc gives a band spectrum more closely resembling that of the sun than the mercury arc which was first considered. Thirty-four percent of the light intensity is of wave length below 4,100 Å°, and none is below 3,100 Å°. <sup>11</sup> In the case of the mercury arc 48 per cent of the intensity is below 4,100 Å° and 36 per cent is below 3,100 Å°, and is therefore not of the same nature as sunlight. However, shorter wave lengths are found more effective in inducing oxidation of oleoresinous films <sup>12</sup> and would doubtless speed up the deterioration of asphalt surfaces more rapidly than the light from the carbon arc. It is doubtful, though, if the deterioration would be as close an approximation to that caused by the natural source.

<sup>11</sup> Nelson, H. A., and Schmutz, F. C., Accelerated weathering: Jour. of Ind. and Eng. Chem. vol. 18, p. 1222, 1926.

<sup>12</sup> Idem.

The lamp was of the automatic feed type, and operated on a line voltage of 110 A. C. The arc voltage was 75 and the current 7 amperes. The carbons burned at the rate of about  $1\frac{1}{2}$  inches per hour from both top and bottom.

The apparatus used in water treating consisted of an ordinary garden hose nozzle attached to a faucet with about four feet of hose. This nozzle was clamped in a vertical position over the center of the container and the stream impinged on a steel plate laid on the perforated disk near its center.

### PREPARATION OF SAMPLES

The types of samples were prepared. Those prepared first were of light weight canvas cloth saturated sewed over wire frames about 3x11 inches and with the bitumen at a temperature of about 200° F. These samples have since proved to be of little value, as no results capable of interpretation can be detected from them.

Samples which consisted of a coating of the bitumen on a steel surface were also prepared. One-inch strap iron was cut into three-inch lengths and a bright surface was ground on one side and the designation was stamped on the other side. Several methods were tried for applying the asphalt, none of which were absolutely successful in securing an even coating uniform in thickness throughout all the samples.

First, attempt was made to paint the material on while in a melted condition. This was unsuccessful as it hardened in the brush almost immediately. Then the steel plates were heated and the material poured on while hot, with the idea of surplus material draining off and leaving an even coating. This was also unsuccessful, as the resulting coatings were too thick and ran off the plates badly under the influence of the heat of the arc. The next method tried was to mix up a thinner paint from the bitumen with a solvent such as CS<sub>2</sub>, CCl<sub>4</sub>, or C<sub>6</sub>H<sub>6</sub>. None of these attempts gave the desired result. The method finally adopted appears the least scientific of those tried, but the results justified it. The plates were heated on a hot plate to 300°F. and a piece of the solid bitumen weighing from 0.5 to 0.7 gm. was held and passed gently back and forth over the hot surface until it had melted into a suitable film. The samples were allowed to remain on the hot plate five minutes in order for any irregularities in the surface to flow out, and were then removed and allowed to cool to room temperature. The resulting surfaces were smooth and black and of a relatively uniform thickness of about 0.01 inch. There was only one sample with which any particular difficulty was experienced. This was one of the foreign native asphalts; the trouble resulted from its high melting point and the presence of sand.



### METHOD OF PROCEDURE

After the samples had cooled, special care being taken to keep them level during the cooling process, they were arranged around the disk in the container equidistant from the center. Here again, care was taken to keep the samples as level as possible. Originally it was tried to place the sample plates perpendicularly around the wall of the container, in order that the surfaces might be at right angles to the arc. This would doubtless have been more satisfactory. This method was found impracticable as the asphalt gradually flowed from the plates while in this position. After spoiling one set of samples it was decided to place them horizontally. The container was put in place and the lamp, suspended from a beam by a wire rope, was lowered through the opening in the cover of the container until the bottom carbon carrier was about one inch above the false bottom. The arc had vertical traveling range of about eight inches and the average distance of the light source from the samples was seven inches.

The maximum temperature attained by the asphalt surfaces was 190° F. and the average temperature during light treatment was about 180°F. This temperature was measured by laying a thermometer with the mercury bulb coated with asphalt directly on the perforated disk holding the samples. The bulb was placed at the same distance from the light source as the test surfaces. The thermometer could be read in place. This temperature is about 30° F. higher than that attained by any black surface directly exposed to the sun on a hot summer day.

Light treatment was given from eight to twelve hours a day<sup>13</sup> until about thirty-five hours of light had been given. Then the container was placed over a sink and the nozzle clamped in place for the water treatment. The impingement of the stream on the metal plate in the bottom finely divided it and made a homogeneous spray throughout the interior of the can. Tap water was used in this treatment. No provision was made for simultaneous light and water treatment.

The water treatment was given in three installments of approximately twelve hours each, between which the light treatment was given. After this the samples were put back on light for 8 to 15 hours a day and idle the remainder of the twenty-four until a total of 160 hours of light had been given. The test was stopped here because not much time for further treatment remained and it was desirable to make a check run. This check was run in the identical manner as the original run described, aside from the fact that the three installments of water treatment were not given until 60 hours of light treatment had been given. At the end of this check run an extra twelve-hour water treatment was given with the

<sup>13</sup> NOTE: The light would run only two hours without adjustment and, therefore, could not be run at night.

idea of inspecting the steel plates for rust after removing the asphaltic coating. This could be taken as a rough measure of the permeability of the film to water.

Furthermore, there had been little change in the appearance of the samples since the point of about 125 hours of light treatment was reached. Further deterioration under treatment is inevitable, however, and in subsequent tests it is recommended that the treatment be carried on about 300 hours. If it had not been felt necessary to make a check run (as it proved not to be, since the checks obtained were remarkable), possibly more comprehensive results could have been obtained. The ratio of water treatment to light treatment is approximately that recommended by Walker and Hickson.<sup>14</sup>

The samples were examined with the naked eye and a low power (30 X) binocular microscope. Microscopic examination was made only at the outset and at the conclusion of the tests, but ordinary observation was made every day or two. Photographs were taken of the original samples before treatment and of the same samples at the conclusion of the test. Also, microphotographs were taken. A general idea of the magnitude of the deterioration can be gained from these but the general appearance is misleading in a number of instances.

### RESULTS

#### GENERAL ACTION OF WEATHERING AGENTS

The common type of failure of the surfaces was a complete dulling of the original luster and the shrinkage of the surfaces into a hard, wrinkled film caused by a combination of oxidation and polymerization of the surface film. See plates I to V. This shrinkage usually accompanied the appearance of broad cracks which were not breaks and did not extend completely through the film on the plates. These "cracks" retained the original luster of the sample in their troughs throughout most of the test, due to new material being drawn into them when the weakened surface was pulled back by the shrinkage of the remainder of the surface of the sample. This effect was more noticeable in the natural asphalts than in the petroleum asphalts, but the latter had a greater tendency to actually crack clear through the film.

As before stated no means were provided for differentiating between the deterioration caused by the light and that caused by the water. The general action of water is to dissolve soluble products and, in the case of soft samples, possibly to emulsify the surface. The erosional effect is probably quite small in any event, and is doubtless small in the case of these tests since no particles

<sup>14</sup> Op. cit.

of water having appreciable size or velocity impinged directly against the test surfaces. The appearance of the samples showed no appreciable change immediately after water treatment, but it was notable and significant that the effect of the light was greatly accelerated immediately succeeding the water treatment. It was for this reason that the water treatment was delayed in the check run, to see if the deterioration would get under way. It did not to any great extent, but after one day of light treatment succeeding the water treatment signs of failure were noted in most of the samples. It would be interesting to make a more comprehensive study of this phase of the problem. It is possible that products formed by original light treatment but remaining in place are washed out by the water, giving subsequent action caused by light the opportunity to alter the appearance of the surface.

#### CONDITION OF INDIVIDUAL SAMPLES

##### SAMPLE A

Most of the original luster is retained. There is no apparent shrinkage or wrinkling of the surface. A slight "hammered" appearance (as hammered silver) is noticeable when the sample is rotated in the sunlight and a network of small elongated depressions, resembling cracks but carrying no actual break in the surface, is apparent on close examination. Under low magnification the surface appears fairly smooth and black, but is minutely pitted.

##### SAMPLE C

The surface is fairly smooth and black. There are spots showing shrinkage in fan shaped areas causing dulling of luster. There are some partings in the surface near these areas having smooth black troughs. Microscopic examination reveals a slight roughness over the surface caused by pitting.

##### SAMPLE D

The luster is dulled and the general surface has a rough appearance. There are several large "vein like" irregular cracks across the surface having troughs of comparatively bright luster but weathering is evident in these. These cracks run at random over the entire surface with rough areas between, in which are indications of new cracks. Under the microscope the roughness seems to be in the form of innumerable peaks and ridges. The nearest reproduction of the appearance is a piece of black grain leather.

##### SAMPLE E

This sample is in bad shape. It looks practically like *D* but is of lighter color. The roughness, as viewed under low power

#### CONDITION OF INDIVIDUAL SAMPLES

magnification, appears to lean more toward ridges and troughs than peaks and pits.

##### SAMPLE F

Practically the same as *E*.

##### SAMPLE G

This surface also has an appearance very similar to *E*. Samples of *D*, *E*, *F*, and *G* are very much alike, and the personal factor would probably determine their place in comparative rating of deterioration.

##### SAMPLE H

The condition of this sample is probably the worst of the lot. The deterioration is of the same general nature as the four preceding samples, but is of slightly higher magnitude. All of the characteristic types of failure are present except actual cracking through the surface. Under low power magnification the surface appears to have a very rough leathery texture. The roughness seems to be due to the shrinkage into peaks surrounded by pits with the tops of the peaks showing small troughs and ridges. The broad smooth-bottomed cracks which run at random over the surface have rough material built up along the edges giving a raised effect to the cracks.

##### SAMPLE I

The surface is relatively bright and in fair shape. A local spot appears to be in excellent condition. However, the entire surface is marked by relatively deep irregular partings or cracks. These occur practically at random over the surface, at distances ranging from  $\frac{1}{4}$  to  $\frac{1}{2}$  inch apart. Magnified, the general background of the surface is smooth and black but is completely covered by a network of small cracks—not deep, and apparently not of the same nature as the broad lustrous cracks on some other samples.

##### SAMPLE L

This sample appears to be in excellent condition. The original luster is still present and the only signs of deterioration are very small "ripple marks". Magnified thirty diameters, some roughness is noted near the edges, and a few scattered eyelet-looking shrinkage areas can be seen.

##### SAMPLE N

The condition of this sample is very good—similar to *L*. The luster is slightly dulled, and the only evidence of weathering dis-

cernable through the microscope appears in a limited area showing roughness of minute design, as small depression rays emanating from points and interlocking.

#### SAMPLE O (Trinidad)

The luster is dulled but the surface is generally smooth. There are several relatively large cracks or partings of the surface, exposing a rough trough. There is an area of general roughness and shrinkage near one end.

#### SAMPLE BER (Bermudez)

This sample withstood the tests very poorly. One area has retained most of its luster near one side, but the remainder of the surface shows shrinkage and wrinkling unequaled in any of the other samples. Some roughness is notable in the smooth area when viewed under a microscope. There are some wide irregular cracks similar to those in sample II.

#### SAMPLE Z (Hard petroleum asphalt)

The general condition is poor. The luster is completely gone and the color changed to dark gray and brown. The entire surface, although generally smooth, shows minute corrugations and a close network of small cracks. These appear as actual cracks, and not just partings in the surface film.

#### SAMPLE P<sub>1</sub>

The surface is generally smooth, but the luster is mostly gone. Noticeable cracking and slight pitting can be seen under the microscope.

#### SAMPLE P<sub>2</sub>

Surface is fairly uniform but hardly any luster is retained. There are no cracks but in some places dull specks appear to be where shrinkage has been followed by the sealing off of a small portion of the surface. Magnified, the surface appears as black grain leather with interwoven shrinkage depressions radiating from pits.

#### SAMPLE P<sub>3</sub>

Fairly regular surface, and some luster retained. A very fine pitting can be detected on close observation. Under the microscope, the pitting is more evident—about like P<sub>1</sub> except there are no cracks.

#### SAMPLE P<sub>4</sub>

Most of the luster is gone and the surface has a slightly corrugated or wavy appearance. There is slight pitting and a general leathery appearance under the microscope.

#### SAMPLE P<sub>5</sub>

This sample is too soft to give very good results. It has retained an appreciable amount of luster but is dulled in spots giving a dappled appearance. The spots when closely examined show to be shrunken areas. The general appearance is smooth and black.

In no case did the plates show any sign of rust when the bitumen was removed.

Table 1.—Comparative classification of samples.

RATING	SAMPLE	NATURE OF ASPHALT	LOCATION
1	L	Natural Oklahoma, Sand.	Johnston County
2	N	Natural Oklahoma, Sand.	M'Curtain County
3	A	Natural Oklahoma, Sand.	Carter County
4	P <sub>5</sub>	Soft Steam refined petrol.	California
5	C	Natural Oklahoma, Sand.	Garvin County
6	I	Natural Oklahoma, Rock.	Murray County
7	P <sub>3</sub>	Air blown petroleum	California
8	P <sub>4</sub>	Steam refined petroleum	California
9	O	Natural	Foreign
10	P <sub>1</sub>	Air blown petroleum	California
11	P <sub>2</sub>	Steam refined petroleum	California
12	D	Natural Okla., Sand & lime	Pontotoc County
13	G	Natural Oklahoma, Sand.	Murray County
14	F	Natural Oklahoma, Rock	Murray County
15	E	Natural Okla., Rock & lime	Murray County
16	Z	Hard Steam refined petrol.	California
17	B	Natural	Foreign
18	H	Natural Oklahoma, Sand	Murray County

The decisions on which the formulation of this table is based were in many cases (in fact in most cases) very close and the personal factor is probably large. It is hard to quantitatively classify failures of different types and this condition obtained in several instances. However, all available data were carefully weighed and it is believed that this rating is fairly correct.

There are several interesting features in Table 1. The most important to the casual observer is the fact that three Oklahoma asphalts head the list. This is not as significant as it might seem, however, since only one of these is from a known large asphalt-bearing

area. Prospecting may prove the Johnston and McCurtain County areas equally productive, but this is doubtful. What is of more significance is the fact that the general standing of the natural Oklahoma asphalts is well above that of the other natural asphalts which furnish a large part of the world's supply. The mean rating of the Oklahoma asphalts is 8.9 (calculated by adding individual ratings and dividing by number of samples), while the mean rating of the foreign natural asphalts is 13.5. The mean rating for the petroleum asphalts is 9.3.

An important, though deplorable observation, is that the asphalts occurring in Murray County in the Arbuckle Region, which is the largest asphalt bearing area in Oklahoma, do not stand up well under the weathering tests. Samples *E*, *F*, *G*, and *H* are all low in the table, and all of these are from that region. Sample *I* is an exception. It comes from a large open rock quarry near Dougherty. The sample to which Abraham<sup>15</sup> refers is from a Carter County deposit, and it is a fact that the Carter County asphalt is placed relatively high in Table 1. This is significant in that it checks Abraham's observation and also important because of the fact that Carter County asphalt is extensive in amount.

Consideration of the positions of the petroleum asphalts is interesting though their limited source materially lessens their value as criteria of comparison. If subsequent work is done along this line it is recommended that samples of petroleum asphalts be obtained from various parts of the country, and that close consideration be given the following discussion before they are used. Samples *P*<sub>1</sub>, *P*<sub>2</sub>, and *Z* are all steam refined asphalts ranging in penetration in the order 141—56—15—2. The positions of occurrence in the table are 4th, 8th, 11th, and 16th, respectively. This seems to indicate an inverse ratio of deterioration to penetration. What makes this even more striking is a consideration of samples *P*<sub>3</sub> and *P*<sub>4</sub>. These are both air blown asphalts having penetrations of 27 and 12 respectively. They occupy positions 7 and 10 in Table 1 in the order named. In other words, here again we find the deterioration or accelerated weathering occurring roughly in an inverse ratio to the penetrations. These, however, do not fit in between the steam refined asphalts in the penetration-weathering relation, but are of a different series.

No such relation as that mentioned above can be established in the case of the natural asphalts. Furthermore, a careful study has been made of the analyses and their relation to Table 1. No conclusion can be drawn. There seems to be no correlation in any instance.

Some relation can be detected between weathering qualities and occurrence. Samples *L*, *N*, *A*, the three best, all occur in the Trinity sand. Sample *C*, which has fair weathering qualities, occurs

in a calcareous sand of the Clear Fork-Wichita formations. Samples *E*, *G*, and *H* occur in the Simpson formation, and their weathering qualities seem to be about the same—all poor. Samples *F* and *I* occur in the Viola limestone. They have weathered differently, however, and do not appear to be classifiable on that basis. It may or may not be significant that the age of the formations in which the asphalt occurs is roughly proportional to the deterioration observed in the samples; that is the younger asphalts withstand weathering better than the older ones.<sup>16</sup>

There is one unfortunate omission in these tests, and that is a sample from the Glenn formation. The representative sample from this formation from a pit northwest of Ardmore was destroyed by accident at a date too late for correction.

### DISCUSSION OF PHOTOGRAPHS

(Plates I to V incl.)

The photographs, in general give some idea of the amount of deterioration but, as said before, they are in some cases misleading. The first section of small photographs are slightly smaller than natural size. They are fair representations of the natural surfaces, but the position and amount of light was not sufficient on it to show up the failings. Also *P*<sub>1</sub> looks very good and *Z* does not look as bad as some of the other samples which are rated higher than it in Table 1. The reason for this is that the photographs do not show up the fine system of cracks which was taken into consideration in the rating.

On the other hand, samples *L* and *N* look not so good as some others in the photographs, but the white specks over these surfaces are due to the material which has been deposited on them from the cored carbons during light treatment. This mineral matter was unavoidably deposited on the samples, and stuck to the softer ones. In general, it makes the samples appear worse in the photographs (especially the microphotographs) than they really are. A close observation of the bitumen on the surface, ignoring the mineral matter, gives the desired information. More definite ideas can be obtained from the description than from the photographs. It is highly improbable that this mineral matter affected the weathering, as it appears highly inert. However, it is recommended that in subsequent tests its deposition on the samples be removed.

The microphotographs are hard to interpret. The magnification is too high and the effect of the mineral matter on the surface too great to gather definite results or be able to correlate any characteristics of the sample with a design of the failure or shrink-

<sup>16</sup> This brings up the question as to whether the oil and asphalts found in the Trinity sandstone were derived from the Pennsylvanian or older rocks buried beneath the Cretaceous overlap, or if they are indigenous to the rocks in which they are found. Preponderance of opinion seems to point to the former possibility. (Editor's note).

age of the surface. It is evident that sample *BER* is in much worse condition than sample *L*, the best according to observation, and also that sample *E* is in worse condition than samples *A* or *I*, but the true condition of the surface cannot be ascertained. The light spots are due to either mineral matter or raised portions due to shrinkage. The regrettable feature is that the failures due to small cracks are not observable. These are evident in sample *Z* on observation, but cannot be seen in the photographs.

### CONCLUSIONS

From the data and discussion in the preceding pages the following conclusions may be drawn:

1. The test method used was successful in that it caused deterioration of the surfaces. However, it would be highly desirable to check the accuracy of the method by natural tests extending over a considerable period of time.

2. Light is the most effective weathering agent, but treatment with water seems to accelerate the effects of the light.

3. Oklahoma asphalts are, in general, better than the other natural asphalts tested. The degree of superiority is not sufficient to warrant any extensive resumption of activity at the present time.

4. Oklahoma asphalts weather in a different manner from the petroleum asphalts tested, but the general magnitude of deterioration is about the same. These tests are not comprehensive, however, since the source of the petroleum asphalt was limited to one locality, and it is highly probable that these asphalts are of an exceptionally good grade.

5. Asphalts occurring in the Trinity sandstone show exceptionally good weathering qualities.

6. Asphalts occurring in the Simpson formation show poor weathering qualities, comparable to the natural foreign asphalts.

7. Petroleum asphalts from a given source, and having been given similar treatment, weather almost directly proportional to hardness.

8. Natural asphalts show no relation between hardness or other analytical data and weathering properties.

Table 2. Per cent bitumen in extracted samples.  
(Extracted with  $\text{C Cl}_4$  by Herbert Peters, Jr.)

Sample	Location	Geologic Formation	Bitumen Per cent.
1	SE. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 10, T. 5 S., R. 1 E.	Deese	8.82
2	NW. $\frac{1}{4}$ SE. $\frac{1}{4}$ Sec. 14, T. 5 S., R. 1 E.	Deese	9.15
3	NW. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 26, T. 5 S., R. 1 E.	Deese	7.75
5	SE. $\frac{1}{4}$ SW. $\frac{1}{4}$ Sec. 16, T. 4 S., R. 1 E.	Deese	8.50
9	NW. $\frac{1}{4}$ Sec. 11, T. 2 S., R. 2 W.	Permian	8.55
13	NW. $\frac{1}{4}$ Sec. 18, T. 3 S., R. 1 E.	Deese (?)	6.57
17(A)	SE. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 18, T. 5 S., R. 1 W.	Permian	13.35
18	SW. $\frac{1}{4}$ Sec. 6, T. 6 S., R. 2 E.	Trinity (?)	8.91
19	NE. $\frac{1}{4}$ SW. $\frac{1}{4}$ Sec. 27, T. 6 S., R. 2 E.	Trinity	12.18
20	NW. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 18, T. 6 S., R. 2 E.	Hoxbar (?)	6.87
21	NE. $\frac{1}{4}$ SE. $\frac{1}{4}$ Sec. 35, T. 6 S., R. 2 E.	Trinity	5.65
23	Sec. 26, T. 4 N., R. 11 W.— $2\frac{1}{2}$ Mi. W. and $\frac{1}{2}$ Mi. North of Elgin	Permian	7.80
26	SW. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 24, T. 2 N., R. 11 W.	Permian	7.69
28	E. side Sec. 27, T. 1 S., R. 5 W.	Permian	9.45
29	SE. $\frac{1}{4}$ Sec. 6, T. 2 S., R. 4 W.	Permian	2.05
31	Center Sec. 25, T. 3 S., R. 5 W.	Permian	8.88
33	NW. $\frac{1}{4}$ Sec. 16, T. 1 N., R. 3 W.	Permian	12.73
34	NW. Corner Sec. 20, T. 1 N., R. 3 W.	Permian	10.51
35	Center Sec. 24, T. 1 N., R. 3 W.	Permian	2.13
36(C)	NW. $\frac{1}{4}$ Sec. 36, T. 1 N., R. 3 W.	Permian	12.31
37	SE. $\frac{1}{4}$ Sec. 29, T. 2 N., R. 6 E.	Simpson (?)	14.26
38(D)	SW. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 36, T. 4 N., R. 5 E.	Ada	6.47
39	NW. $\frac{1}{4}$ Sec. 31, T. 4 N., R. 6 E.	Ada	4.97
40	SW. $\frac{1}{4}$ Sec. 30, T. 4 N., R. 6 E.	Ada	4.68
41	NE. NW. 28 & SE. SW. 21, T. 1 N., R. 4 E.	Simpson	10.90
42(E)	SW. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 14, T. 1 S., R. 3 E.	Simpson	10.74
43	SE. $\frac{1}{4}$ Sec. 23, T. 1 S., R. 3 E.	Simpson	12.96
44(F)	S. $\frac{1}{2}$ SW. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 22, T. 1 S., R. 3 E.	Hunton (?)	5.83
45	SW. $\frac{1}{4}$ NE. $\frac{1}{4}$ Sec. 22, T. 1 S., R. 3 E.	Simpson	6.37
47	SW. $\frac{1}{4}$ SE. $\frac{1}{4}$ Sec. 15, T. 1 S., R. 3 E.	Simpson	8.89
48	NW. $\frac{1}{4}$ Sec. 22, T. 1 S., R. 3 E.	Simpson	9.37
49(G)	NW. $\frac{1}{4}$ SE. $\frac{1}{4}$ Sec. 22, T. 1 S., R. 3 E.	Simpson	9.37
50, 51, 52	SE. $\frac{1}{4}$ Sec. 21, T. 1 S., R. 3 E.	Simpson	6.38
53	SE. $\frac{1}{4}$ Sec. 17, T. 1 S., R. 3 E.	Simpson	6.99
56(H)	NE. $\frac{1}{4}$ NW. $\frac{1}{4}$ Sec. 29, T. 1 S., R. 3 E.	Simpson	9.57
57(I)	SE. $\frac{1}{4}$ Sec. 25, T. 1 S., R. 2 E.	Viola	4.25
59	Center Sec. 21, T. 1 S., R. 2 E. ?	Viola	0.62
60	NW. $\frac{1}{4}$ SE. $\frac{1}{4}$ Sec. 29, T. 4 S., R. 8 E.	Trinity	4.90
61	SE. $\frac{1}{4}$ Sec. 19, SW. $\frac{1}{4}$ Sec. 20, T. 4 S., R. 6 E.	Trinity	4.23
63	NE. $\frac{1}{4}$ Sec. 14, T. 4 S., R. 5 E.	Trinity	2.45
64	NE. $\frac{1}{4}$ NW. $\frac{1}{4}$ Sec. 20, T. 5 S., R. 5 E.	Trinity	2.75
67	SW. $\frac{1}{4}$ Sec. 32, ?	Trinity	2.67
70	NW. $\frac{1}{4}$ Sec. 26, T. 7 S., R. 5 E.	Trinity	4.56
73(L)	SW. $\frac{1}{4}$ Sec. 27, T. 4 S., R. 8 E.	Trinity	9.32
76	SE. $\frac{1}{4}$ Sec. 24, T. 4 S., R. 9 E.	Trinity	6.82
78	About Sec. 24, T. 3 S., R. 11 E.	Trinity	5.73
89	SE. $\frac{1}{4}$ Sec. 2, T. 2 N., R. 19 E.—in Potato Hills	Stanley	0.73
95	Sec. 20, T. 7 S., R. 24 E.	Trinity	10.39
98	Valliant Deposit	Trinity	12.35
99(N)	Valliant Deposit	Trinity	11.47

Table 3.—*Chemical Properties of Oklahoma Asphalts*

Tests made at 77° F.

## WEATHERING PROPERTIES OF OKLAHOMA ASPHALTS

SAMPLE	SOLUBLE IN CS <sub>2</sub> PER CENT	BITUMEN INSOLUBLE IN PETRO- LEUM ETHER PER CENT	CONSIS- TENCY	SPECIFIC GRAVITY	DUCTILITY	MELTING POINT °F	PENETRA- TION cm.	HARDNESS
17(A)	99.30	35.10	36.1	1.04	4.5	170†	.....	.....
23	99.6	11.9	0.0	0.97	*	*	.....	.....
36(C)	99.9	28.0	20.0	1.03	Over 100	140	2.9	.....
38(D)	99.0	25.5	8.88	1.02	*	121	.....	.....
42(E)	99.2	23.8	7.37	1.02	*	116	.....	.....
44(F)	99.5	15.61	4.64	1.02	*	*	.....	.....
49(G)	99.4	16.83	5.19	1.02	*	113	.....	.....
56(H)	99.9	25.13	8.43	1.03	Over 100	117.5	.....	.....
57(I)	99.4	24.83	8.24	1.035	*	118	.....	.....
61	99.6	12.3	0.0	1.015	*	*	.....	.....
67	99.7	12.9	0.0	1.01	*	*	.....	.....
73 (L)	99.6	15.2	.....	1.025	*	100.4	.....	2.75
76	99.5	13.27	0.0	1.00	*	*	.....	.....
98	99.35	30.6	18.2	1.026	11.5cm.	156	.....	.....

†—Ball and ring.

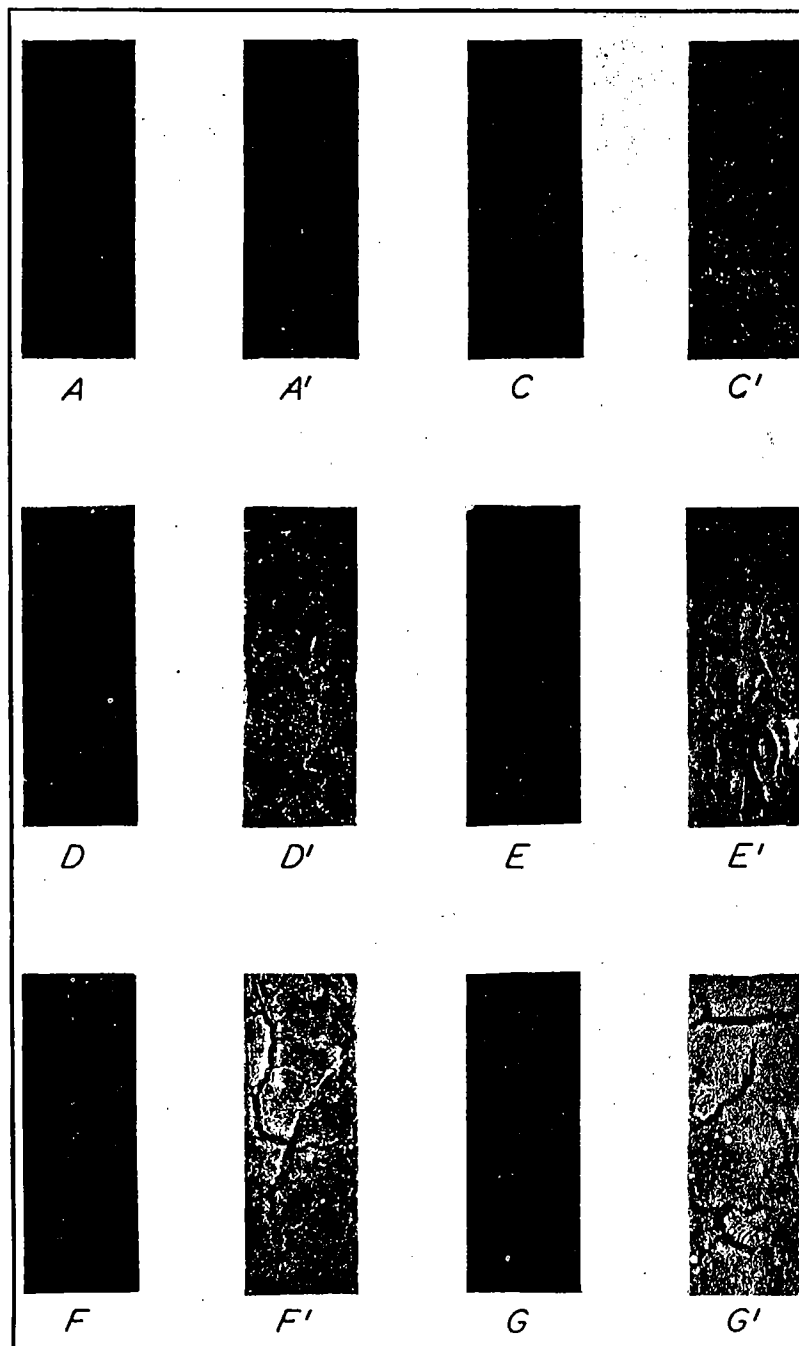
\*—Too soft.

## EXPLANATION OF PLATE I.

## Figure

- A. Sand asphalt, Clear Fork-Wichita formation, sec. 18, T. 5 S., R. 1 W.
- C. Sand asphalt, Enid (Garber ?) formation, sec. 36, T. 1 N., R. 3 W.
- D. Sand asphalt, Ada formation, sec. 36, T. 4 N., R. 5 E.
- E. Sand and lime asphalt, Simpson formation, sec. 14, T. 1 S., R. 3 E.
- F. Limestone asphalt, Hunton (?) formation, sec. 22, T. 1 S., R. 3 E.
- G. Sand asphalt, Simpson formation, sec. 22, T. 1 S., R. 3 E.

Prime (') figures represent samples after weathering tests.  
All figures x.

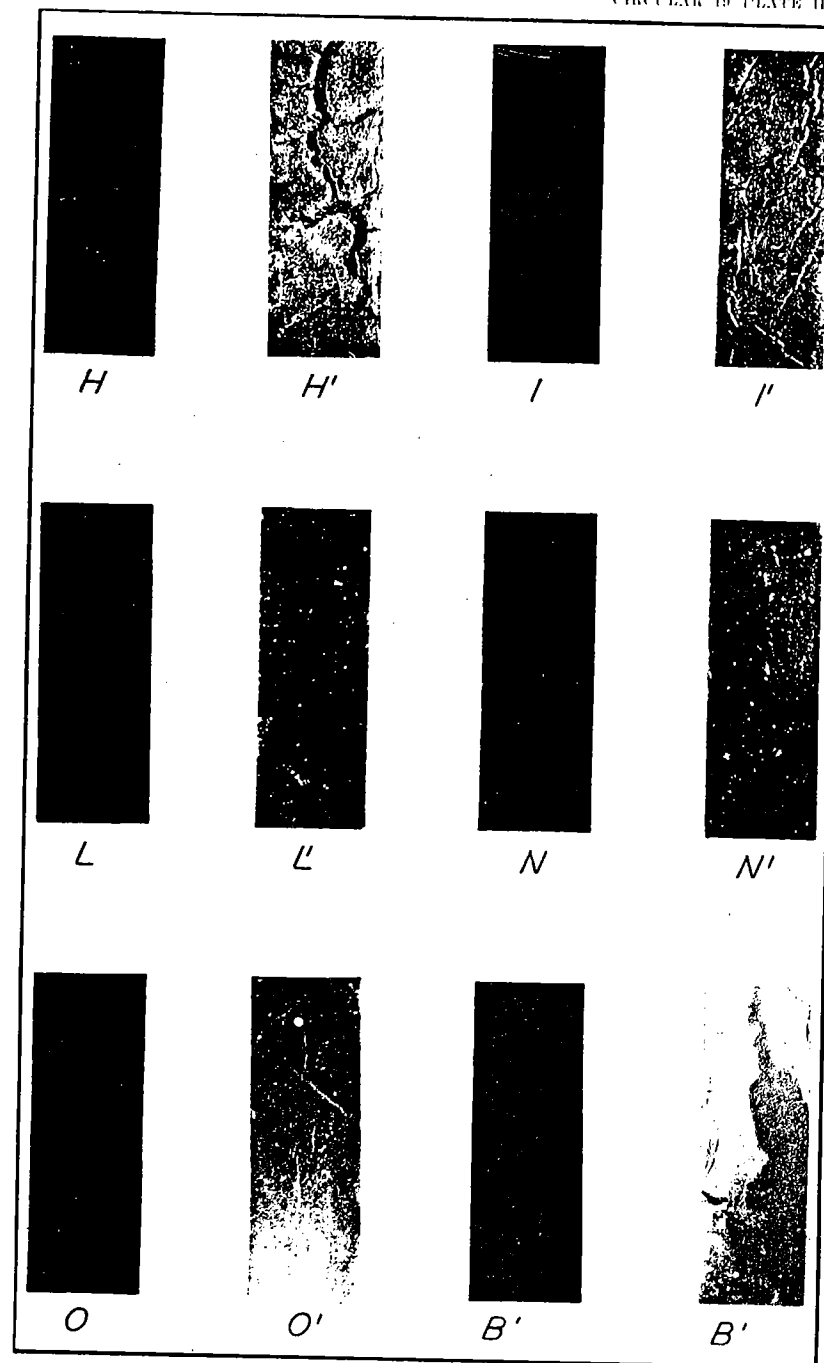


## EXPLANATION OF PLATE II.

## Figure

- H. Sand asphalt, Simpson formation, sec. 29, T. 1 S., R. 3 E.  
 I. Limestone asphalt, Viola limestone, sec. 25, T. 1 S., R. 2 E.  
 L. Sand asphalt, Trinity sandstone, sec. 27, T. 4 S., R. 8 E.  
 N. Sand asphalt, Trinity sandstone, ½ mile east of Valliant, McCurtain County.  
 O. Natural foreign asphalt.  
 B. Natural foreign asphalt.

Prime (') figures represent samples after weathering tests.  
 All figures x.





## EXPLANATION OF PLATE III.

## Figure

Z. Hard steam refined petroleum asphalt.

P<sub>1</sub>. Air-blown petroleum asphalt.

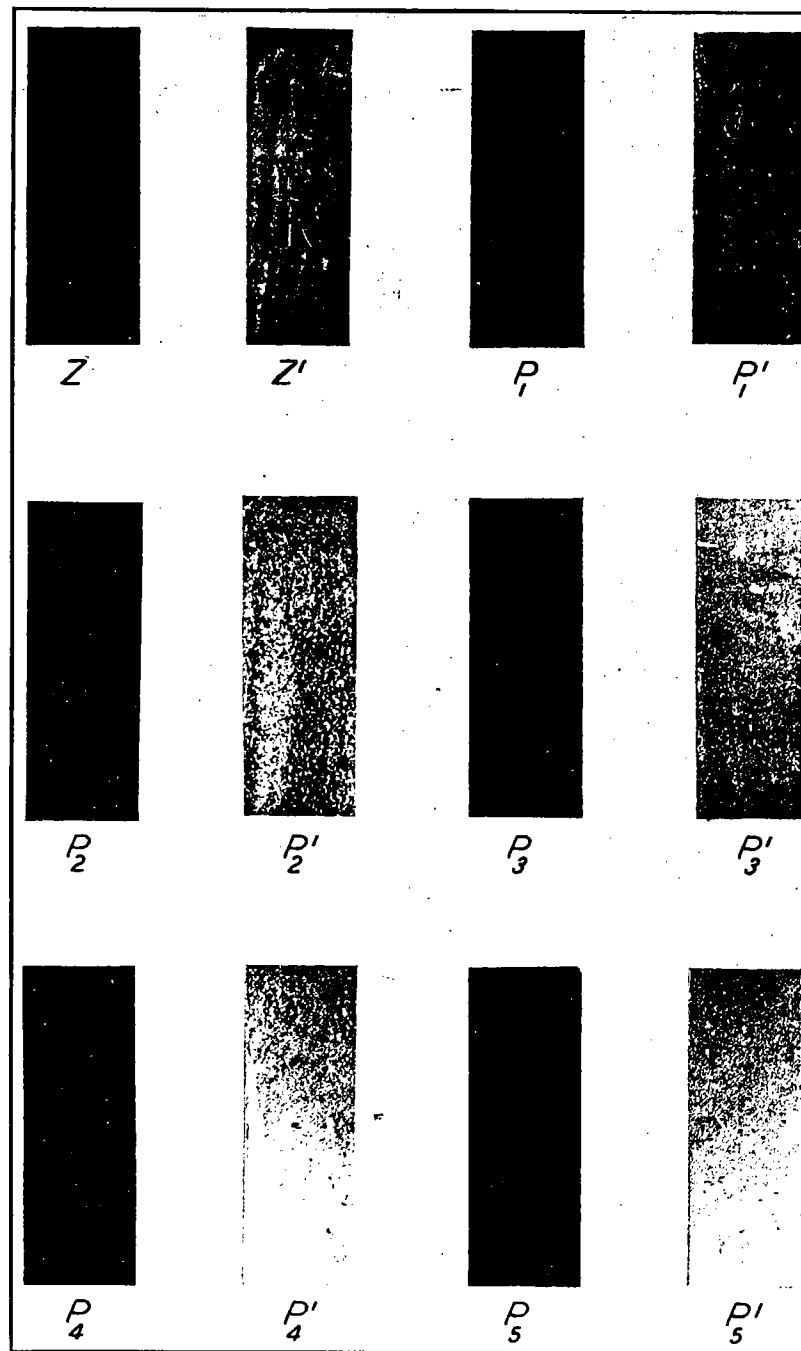
P<sub>2</sub>. Steam refined petroleum asphalt.

P<sub>3</sub>. Air-blown petroleum asphalt.

P<sub>4</sub>. Steam refined petroleum asphalt.

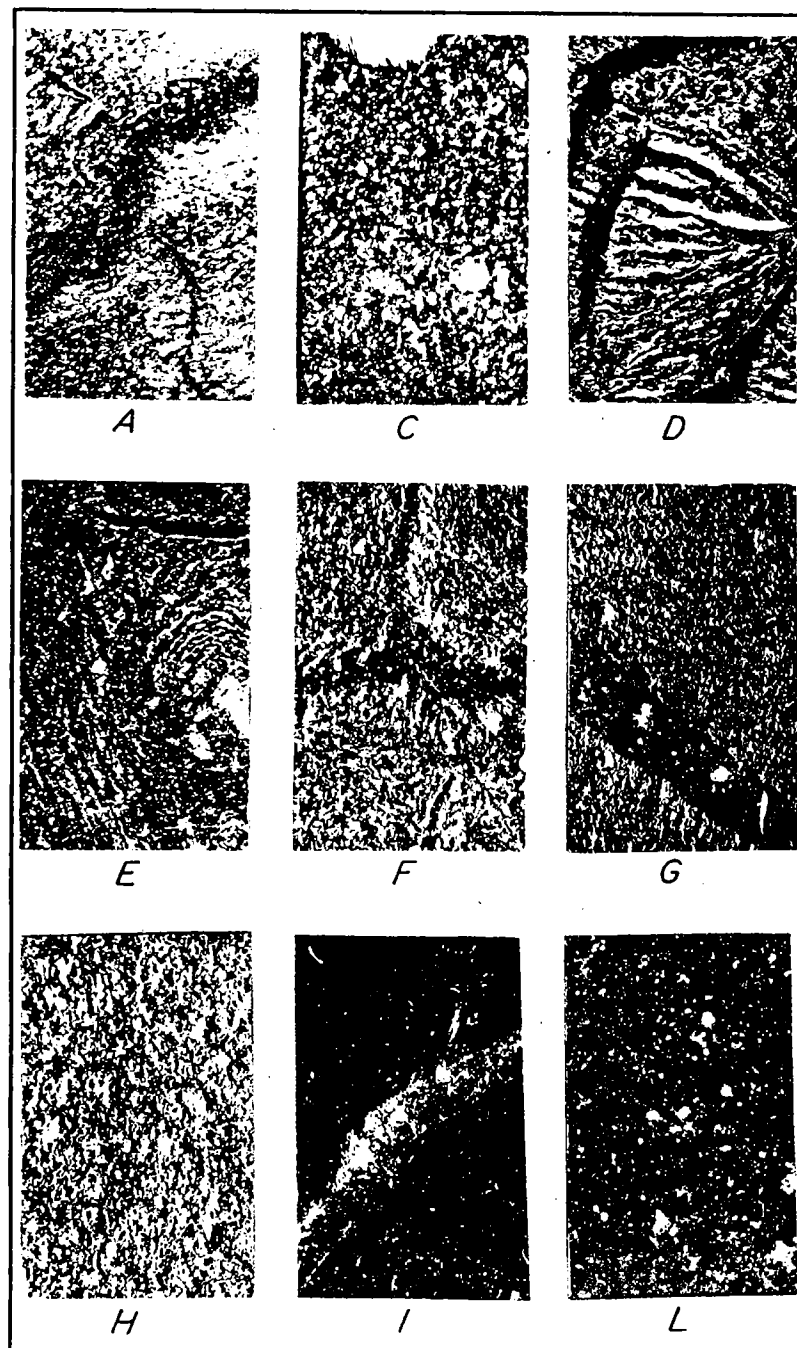
P<sub>5</sub>. Soft steam refined petroleum asphalt.

Prime (') figures represent samples after weathering test.  
All figures x.



## EXPLANATION OF PLATE IV.

Enlarged photographs of samples after weathering. (x10).  
For location and description of samples see Plates I and II.



## EXPLANATION OF PLATE V.

Enlarged photographs of samples after weathering. (x 10).  
For location and description of samples see Plates II and III.

