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ON THE SKY LINE DRIVE IN VIRGINIA

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# PUBLIC ROADS

▶▶▶ *A Journal of Highway Research*

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BUREAU OF PUBLIC ROADS

G. P. St. CLAIR, *Editor*

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# A STUDY OF SOME LIQUID ASPHALTIC MATERIALS OF THE SLOW-CURING TYPE

BY THE DIVISION OF TESTS, BUREAU OF PUBLIC ROADS

Reported by R. H. LEWIS, Associate Chemist, and W. O'B. HILLMAN, Junior Highway Engineer

THERE has been considerable progress in the simplification of tests and specifications for liquid asphaltic road materials, but there is still considerable difference of opinion as to the laboratory tests most highly indicative of the probable road or service behavior of liquid materials of the so-called slow-curing type.

## PRESENT TESTS UNSATISFACTORY AS A BASIS FOR PREDICTING SERVICE BEHAVIOR OF SLOW-CURING MATERIALS

Unlike materials of the medium-curing and rapid-curing types, which are generally combinations of refined asphalts and more or less volatile solvents, and which in the laboratory tests readily lose these solvents, leaving residues possessing essentially the characteristics of the base asphalts, materials of the slow-curing type are of variable composition. They may be crude or reduced petroleum from various fields, blends of topped and residual petroleum with cracking coil residues, cracking coil residues of satisfactory fluidity, and blends of various residuals of widely different consistencies with more or less nonvolatile distillates.

It is still an open question as to what tests and specifications will adequately control and describe the most essential qualities of this type of liquid asphaltic road material. Many of those engaged in the testing and use of these products favor such tests as those for specific gravity, solubility in naphtha, fixed carbon, asphaltic residue and other tests less generally known. Others consider that only the original consistency and an accelerated evaporation test, with a purity and consistency test on the residue, are essentially important in judging or anticipating the road making qualities of a particular fluid product. An accelerated evaporation test is assumed to indicate in a relatively short time the amount of volatile matter lost in a long period of service and the probable consistency of the resulting residue. The accelerated evaporation tests most commonly used are the distillation tests, and the volatilization test or loss on heating at 325° F. for 5 hours.

Slow-curing materials, when subjected to either of these accelerated evaporation tests, yield residues which are fluid and not adapted to the usual tests for semisolid asphalts as are the residues from rapid-curing and medium-curing products. In order to determine the amount of semisolid asphalt which a given liquid asphaltic material may ultimately produce, and in many cases to ascertain the physical and chemical characteristics of this residue by tests usually made on semisolid asphalts, the test for residue of 100 penetration has been used by some testing engineers.

Slow-curing materials are used extensively as dust palliatives, in penetrative treatments such as oiling earth roads, in the so-called blotter treatments and in the oil-processed roads of the West. In these types of surface, the bituminous material is not directly exposed to the action of sun and air in any appreciable thickness, and for this reason it is held by some that if the liquid

asphaltic material, when subjected to either the volatilization test or the distillation test, yields a fluid residue, then the material in the road will probably remain fluid. Others believe that the bituminous material does eventually become semisolid and that the characteristics of the residue of 100 penetration are not only typical of the asphaltic residue which ultimately forms the bituminous binder in the road surface but are also indicative of the serviceability of the original material as a road binder.

Slow-curing materials are altered in service by the loss of volatile matter and also by other factors. The residues obtained in the various laboratory evaporation tests may or may not be typical of service residues. Two slow-curing materials having identical test values, which initially function satisfactorily as dust palliatives, may show great differences in length of efficient service. Two products used in oil processed construction may develop smooth well bonded surfaces when first compacted, yet one may produce a road which can be successfully reprocessed after a long period and the other may produce a surface which rapidly hardens and ravel, requiring the use of additional oil during reprocessing. Many engineers, therefore, have developed tests and specifications which they hope will insure the purchase of liquid asphaltic materials from a particular field or process of manufacture which rendered adequate and economical service. These same tests and specifications may not guard against improper materials when the purchaser is dependent on products from another field or different process of manufacture.

Practically all specifications for slow-curing liquid asphaltic materials require some sort of accelerated evaporation test. The question therefore arises as to whether the accelerated laboratory evaporation tests do indicate the probable road behavior. Like most materials, whether organic or inorganic, bituminous materials suffer change and in many cases complete disintegration on exposure to the elements. Liquid asphalts of the kind considered, used to form a thin coat on mineral aggregate in the exposed surface of the road, no doubt undergo more severe changes on exposure than do the more carefully refined and more viscous asphaltic materials. It seems desirable that changes in physical and chemical characteristics which occur when slow-curing products are exposed to sunlight, heat, and air be studied and compared with the changes produced in the various laboratory tests.

## SLOW-CURING MATERIALS SUBJECTED TO LABORATORY TESTS AND EXPOSURE TO SUN

With this end in view, 22 samples of liquid asphaltic products of the slow-curing type were selected and given a complete laboratory examination, consisting of all tests generally used in the control of materials of this kind. Samples were then exposed in films one-eighth inch thick to the action of air, light, and solar heat during the summer months of 1932. The loss of

volatile matter was determined and the residues, together with the residues from the laboratory evaporation tests, were subjected to determinations which would indicate the extent of the physical and chemical changes occurring under both conditions.

In order to determine the binding value of the original materials and the residues obtained from various laboratory tests Hubbard-Field stability cylinders were prepared with a standard sand and the resulting cylinders tested for stability. Cylinders prepared with the original materials were also exposed to oven heat at 140° F. and to the same exposure conditions as the thin films. At stated intervals the loss of volatile matter and the stability were determined.

The samples selected for this study, with few exceptions, represent materials which will meet one of the provisional specifications for slow-curing liquid products recently developed cooperatively by the Bureau of Public Roads and the Asphalt Institute. They are the products of 10 refineries in the Far West and Middle West and, in most cases, will meet some of the specifications of the States dependent on these refineries for asphaltic materials for low-cost road construction. The samples are identified in table 1, which shows the State specifications which these materials were designed to meet and the cooperative specifications which they will meet.

The laboratory tests on the original materials were as follows:

- Specific gravity at 77° F./77° F.
- Flash point, ° F.
- Saybolt-Furol viscosity at 122° F. and 140° F.
- Float test at 77° F.
- Volatilization at 325° F., 20 and 50 grams.
- Residue, float at 122° F.
- Distillation:
  - Residue:
    - Float at 122° F.
    - Solubility in carbon tetrachloride.
  - Distillate:
    - Specific gravity at 77° F./77° F.
    - Index of refraction at 77° F.
- Residue of 100 penetration:
  - Penetration at 77° F. and 32° F.
  - Ductility at 77° F. and 34°-35° F.
  - Softening point, ° F.
- Fixed carbon.
- Solubility in 86° B. naphtha.
- Solubility in carbon tetrachloride.
- Solubility in carbon disulphide (in some instances).

The test for residue of 100 penetration or asphalt-content test will hereafter be called, as a matter of convenience, the asphaltic residue test, and the residue therefrom the asphaltic residue.

Tests were made, unless otherwise stated, in accordance with the standard or tentative standard methods of the American Society for Testing Materials or the American Association of State Highway Officials and the serial designations of these methods are given in table 2.

The distillation test was made in accordance with the A.S.T.M. Standard Method D 20-30, with the following exceptions:<sup>1</sup>

<sup>1</sup> This is substantially the method recommended by the Bureau of Public Roads and the Asphalt Institute in the proposed simplified scheme of analysis, differing only in the amount of sample distilled, 200 cubic centimeters at 77° F. instead of 60° F., and in the receptacle for the residue. The 400 cubic centimeter beaker was used instead of the 8-ounce tin because the entire contents of the distillation flask could not be poured into the 8-ounce tin.

TABLE 1.—Designation of samples and specification intended to be met

Bureau of Public Roads laboratory no.—	Identification	Producer	Refinery	Specification intended to be met	Meets Bureau of Public Roads and Asphalt Institute provisional specifications
35100	A	1	1	Illinois E-3	
35103	B	1	1	Illinois E-4	S. C.-2
35364	C	1	2	Illinois E-3	
35367	D	1	2	Illinois E-4	S. C.-2
35157	E	2	3	Northwestern Conference No. 2	S. C.-2
34263	F	2	4	Middle Western States 60-70	
34453	G	3	5		
34454	H	3	5	Northwestern Conference No. 2	S. C.-2
34322	I	4	6	Middle Western States 70-80	S. C.-2
34323	K	4	6	Middle Western States 70-80	S. C.-3
36310	L	5	7	Northwestern Conference No. 2	S. C.-2
36303	M	5	7	Colorado 60-70	
36300	N	5	7	California and New Mexico 70-80	S. C.-3
35225	O	6	8	Northwestern Conference No. 2	S. C.-2, 3
35226	P	6	8	Arizona	S. C.-2
35228	R	6	8	California and New Mexico 70-80	S. C.-3, 4
36106	S	5	9	Colorado 60-70	
36089	T	5	9	Northwestern Conference No. 2	S. C.-2, 3
36091	V	5	9	California and New Mexico 70-80	S. C.-3
35077	X	7	10	Arizona	
35080	Y	7	10	Northwestern Conference No. 2	S. C.-2
35081	Z	7	10	California and New Mexico 70-80	S. C.-3

TABLE 2.—Designations of test methods used for various tests

Test	A.S.T.M. serial no.	A.A.S.H.O. serial no.
Flash point, Cleveland open cup	D 92-24	T-48
Specific gravity	D 70-27	T-43
Furol viscosity	D 88-30	T-72
Float test	D 139-27	T-50
Penetration	D 5-25	T-49
Softening point, ring and ball	D 36-26	T-53
Ductility	D 113-26T	T-51
Loss at 325° F.	D 6-30	T-47
Residue of 100 penetration	D 243-28T	
Soluble in CS <sub>2</sub>	D 4-27	T-44
Soluble in CCl <sub>4</sub>		T-45
Insoluble in 86° B. naphtha		T-46
Fixed carbon	D 168-30	

<sup>1</sup> In A.S.T.M. standard method of test D 6-30 there is no provision for making the test with a 20 gram sample.

1. The sample distilled was 200 cubic centimeters at 77° F.
2. The bulb of the thermometer was immersed to a point one-quarter inch above the bottom of the flask.
3. The condenser was water cooled.
4. The distillate was collected in 100 cubic centimeter glass cylinders and the initial boiling point and the percentage of distillate by volume at 680° F. and every 18° F. below 680° F., was recorded. The weight of the total volume of distillate to 680° F. was also recorded.
5. The distillation was stopped at 680° F. and the entire residue poured immediately into 400 cubic centimeter pyrex beakers and cooled to room temperature. After weighing it was then warmed, thoroughly stirred and poured into covered containers for further tests.

The weight of the residue from distillation remaining in the flask after pouring was also determined. The weight of the sample minus the weight of the distillate and residue is the distillation loss. The distillation loss is relatively high because, when the residue is poured at the high temperature of 680° F. considerable volatile matter escapes during the pouring and cooling to room temperature. This loss is reported as loss on cooling. The percentage of distillate collected plus the percentage of loss on cooling is the total percentage

TABLE 3.—Tests on the original materials

Table with 20 columns: Identification, Specific gravity, Flash point, Float at 77° F., Viscosity (Furoil viscosity at 122° F. and 140° F.), Volatilization (50 grams and 20 grams), Organic matter insoluble in CS2 and CCl4, Organic matter insoluble in 86° B. naphtha, Fixed carbon, Percent, Time of reduction, Penetration (77° F. and 32° F.), Softening point, and Ductility (77° F. and 34-35° F.). Rows A-Z.

Table with 15 columns: Identification, Initial boiling point, Total percent off by volume at (500° F. to 680° F.), Total distillate by weight, Loss on cooling, Residue (Float at 122° F., Organic matter insoluble in CS2 and CCl4), and Distillate (Specific gravity and Index of refraction). Rows A-Z.

of loss by weight in the distillation test, and permits a better comparison of the distillation residue with residues from other tests. The specific gravity of the distillate collected was determined by the pycnometer method. The index of refraction at 77° F. was determined by means of a Zeiss refractometer. The results of the laboratory tests are given in table 3 and when possible are shown graphically.

The correlation of test data on asphaltic material in general, and liquid asphaltic products of the slow-curing type in particular, is made rather difficult because of the great variety of petroleum and processes used in their manufacture. A careful analysis of the data will be of interest, especially to those who are not familiar with products from widely different sources.

MATERIALS OF HIGH SPECIFIC GRAVITY SHOW SPECIAL CHARACTERISTICS

In general the test results show that the flash point, consistency, percentage of matter insoluble in naphtha, percentage of fixed carbon and percentage of asphaltic residue of products from the same refinery vary directly as the specific gravity, and the amount of volatile matter varies inversely as the specific gravity. However, these relations are by no means true for the products as a whole. There are certain characteristics which products from different refineries have in common.

Seven of these materials, representing products from four different refineries, have similar characteristics. All have specific gravities greater than 1.000; samples

A and B are only slightly greater, and samples F, G, H, I, and K considerably greater. All of these materials have greater percentages of matter insoluble in naphtha and greater percentages of fixed carbon than the low-gravity products. They are the only materials having an appreciable amount of matter soluble in carbon disulphide but insoluble in carbon tetrachloride (carbenes). Their distillates from the distillation test have high specific gravities and high indexes of refraction. Their asphaltic residues, except those of samples A and B, have the lowest penetrations at 32° F., indicating a high susceptibility to alteration with temperature change and, when tested for ductility at 34–35° F., are brittle and nonductile. Lack of ductility at low temperatures is not, however, a property solely of the asphaltic residues of the high-gravity materials since the residues of some of the low-gravity materials are also non-ductile at 34–35° F. These high-gravity materials are so uniformly different from the other materials included in the series that they are readily identified on most of the graphs as a group.

The results of the consistency tests on the original materials plotted in figure 1, show that the high-gravity materials, except A and B, have lower Furol viscosity values at 140° F. and higher float-test values at 77° F. than the low-gravity materials of the same Furol viscosity at 122° F. It is apparent that the consistencies of these high-gravity materials are more affected by changes in temperature than are the consistencies of the low-gravity materials.

RESULTS OF LABORATORY HEAT TESTS DISCUSSED

Since either 20- or 50-gram samples in tins of standard size are used in the volatilization test, or, as it is sometimes called, the oven test, the materials under investigation were tested by both methods. Figure 2 shows the percentage of loss of 20-gram and 50-gram samples heated for 5 hours at 325° F. and the results of the float test on the residue at 122° F. While the percentage of loss and the float test results on the residues are always higher for the 20-gram samples, there is almost a straight line relation between the percentages of loss and very little relation between the float-test results on the residues.

The results of distillation tests are compared with those from oven tests in figure 3. In the routine performance of the distillation test the percentage of distillate by volume is determined and the volatile matter lost in pouring the residue is not considered. A consistency test on the residue obtained by this method does not represent the consistency of the original material less the volume distilled. Since the weight of distillate lost on cooling was determined, a comparison can be made of the percentage of loss in the volatilization test with the percentage of distillate by volume and with the percentage of total loss in the distillation test by weight, as well as a comparison of the consistencies of the two residues.

In figure 3-A, the percentage of distillate by volume is compared with the percentage of loss by weight at 325° F. for 20-gram samples. The percentage of distillate is usually the greater. Since the distillates are of different densities and since the total percentage off at 680° F. does not include the loss on cooling, which is variable, these percentages of volatile matter, one by weight and the other by volume, are not strictly comparable. Therefore, figure 3-B which compares the

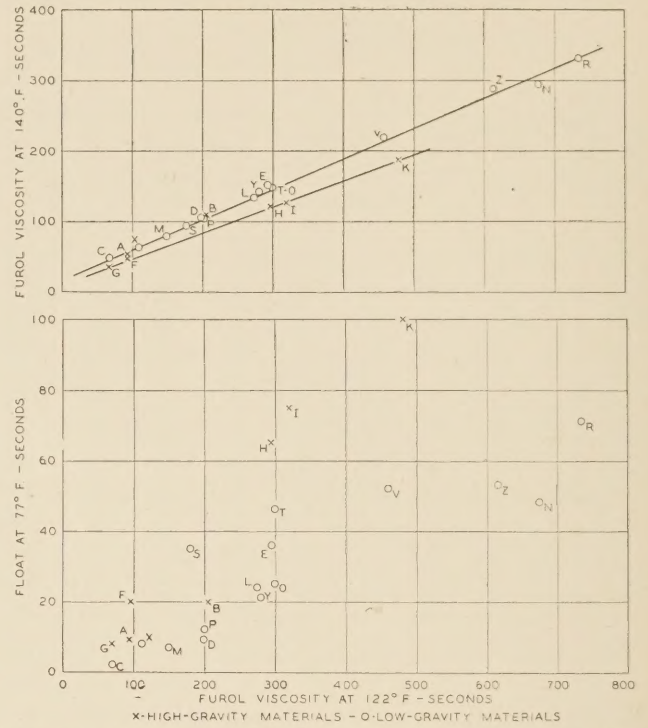


FIGURE 1.—COMPARISON OF RESULTS OF CONSISTENCY TESTS ON ORIGINAL MATERIALS.

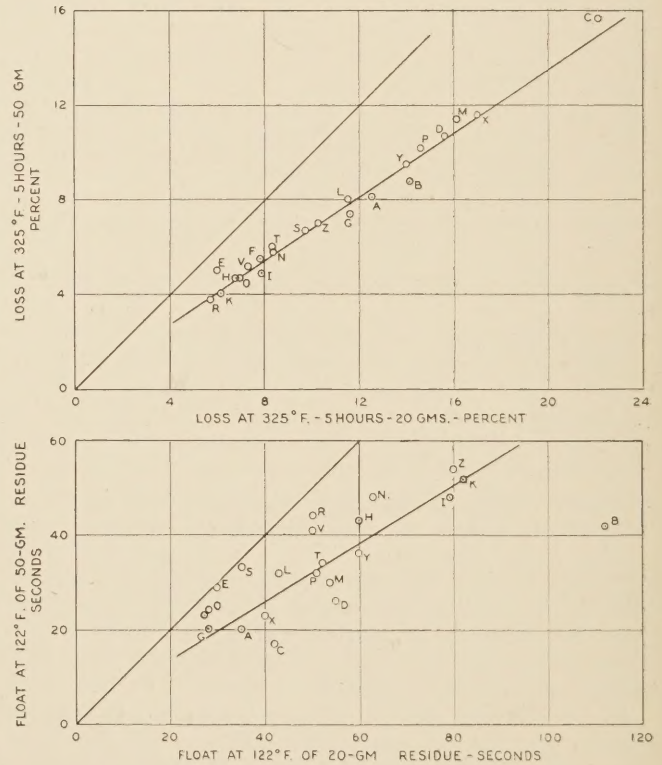


FIGURE 2.—COMPARISON OF RESULTS OF VOLATILIZATION TESTS ON 20-GRAM AND 50-GRAM SAMPLES AND RESULTS OF FLOAT TESTS ON RESIDUES.

total percentage of loss by weight in the distillation test with the percentage of loss by weight in the 20-gram volatilization test gives a truer picture of the relationship between the two tests and shows that a greater loss of volatile matter occurs in the distillation test. This is



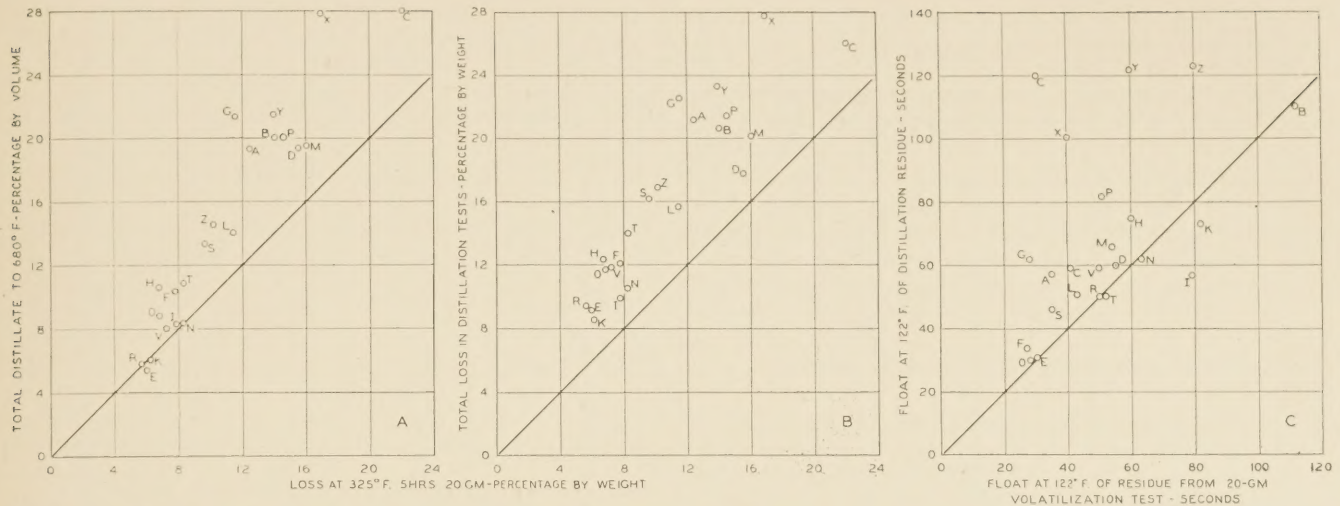


FIGURE 3.—COMPARISON OF THE PERCENTAGES OF LOSS AND THE CONSISTENCIES OF THE RESIDUES FROM THE 20-GRAM VOLATILIZATION TEST AND THE DISTILLATION TEST.

attributed chiefly to the high temperature to which the distillation test is run and the disturbance of the residue in pouring. Figure 3-C shows that in a few cases the volatilization residue has the higher float-test value, although in general the distillation residue is harder.

The distillation curves of these samples, shown in figure 4, clearly demonstrate that they are composed essentially of high boiling constituents, only samples C and D having distillate boiling under 500° F. All of the curves except those for samples C and D are of the same trend, showing a steady rise from the initial boiling point to the end point of 680° F. The curves for samples C and D, however, begin to flatten out at the higher temperatures, indicating comparatively less material boiling immediately above 680° F. The low loss on cooling which occurred when the residue of these two materials was poured is a further indication that there would be less boiling immediately above 680° F. If these liquid asphaltic products harden only through loss of volatile matter and if the consistency of the distillation residue is representative of the ultimate consistency of the material in service, samples C and D are certainly the most rapid hardening. However, if the ultimate consistency in service is appreciably harder than the consistency of the distillation residues, samples C and D will probably take longer to reach their ultimate consistency than the other materials which do not have such a rapid initial loss of volatile matter.

Both specific gravity determinations and index of refraction determinations were made on the distillates. The gravities and indexes of refraction of the distillate from the high-gravity products are shown in figure 5 to be not only generally higher than the low-gravity products but to fall on different curves, indicating that the chemical structure of the distillates of the high-gravity materials is quite different from that of the distillates of the low-gravity materials.

The asphaltic residue test has been and still is used by many of the States for the control of slow-curing materials. The time of reduction is considered by some as an indication of the relative speed at which the volatile matter is lost. Table 3 shows that the time of reduction varied from 25 minutes for samples K and Y to a maximum of 420 minutes for sample D with an average for the entire group of approximately 95 minutes, although only 5 samples took more than 100 minutes.

FOR SLOW-CURING MATERIALS FROM THE SAME SOURCE, THE PERCENTAGE OF ASPHALTIC RESIDUE VARIES DIRECTLY AS THE VISCOSITY AND INVERSELY AS THE PERCENTAGE OF DISTILLATE

In figure 6 the percentage of asphaltic residue is plotted against the Furol viscosity at 122° F. and the percentage of distillate by volume. While there is again the objection to comparing percentage by volume and percentage by weight, the curves shown would not be materially different if either the percentage of loss in the volatilization test or the total percentage of loss by weight in the distillation test were substituted for the percentage of distillate by volume. Since laboratories using the modified distillation test are reporting percentages by volume, this value is used in the comparison.

For the materials as a group there is no direct relation; but if the results for products from the same refinery are joined by straight lines, it is seen that as the asphaltic residue increases, the viscosity increases and the percentage of distillate decreases. Samples M, P, and B are exceptions to this general rule. Samples M and P were made to meet a specification having an exceptionally low viscosity requirement for a given asphaltic residue and do not correspond with the other samples from the same refinery. These two have properties similar to those of samples X, Y, and Z from another refinery. Sample B, although having a higher asphaltic residue and a higher viscosity than sample A, also has a greater percentage of distillate.

A comparison of the curves of figure 6 shows that for the samples as a whole those products having the lowest viscosity for a given percentage of asphaltic residue generally have the highest percentage of distillate. If the time of float of the distillation residue and the time of reduction to an asphaltic residue of 100 penetration are compared with the positions of the materials in figure 6 it will be seen that, except for samples C and D, those products which have the highest percentage of distillate for a given asphaltic residue have, in general, the highest float of the distillation residue and take the shortest time to come to 100 penetration. However, samples C and D, which the distillation curves show to have high initial volatility, are exceptions to this rule taking by far the longest time for reduction to an asphaltic residue of 100 penetration.

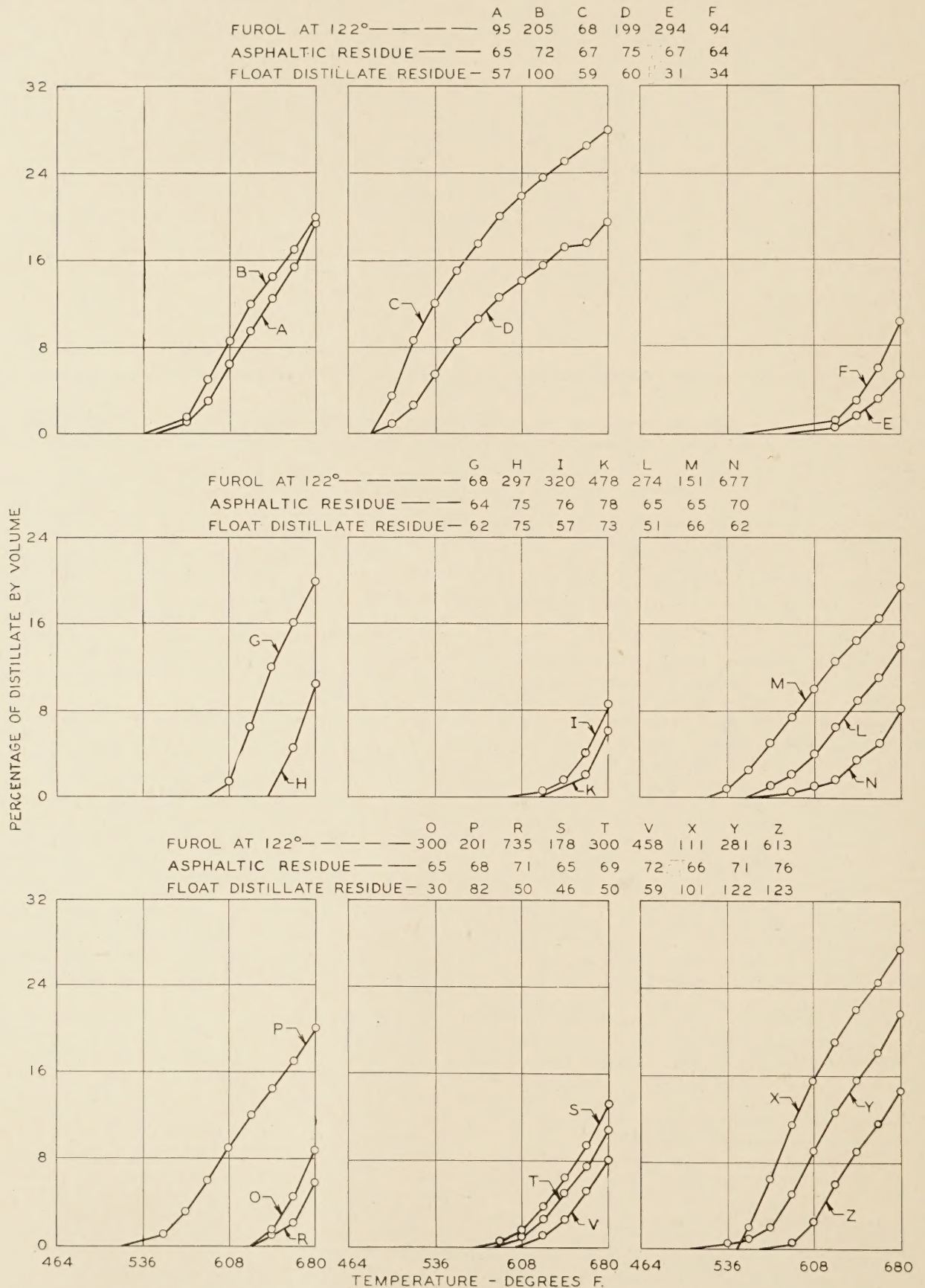


FIGURE 4.—RELATION OF PERCENTAGE OF DISTILLATE BY VOLUME TO DISTILLING TEMPERATURE.

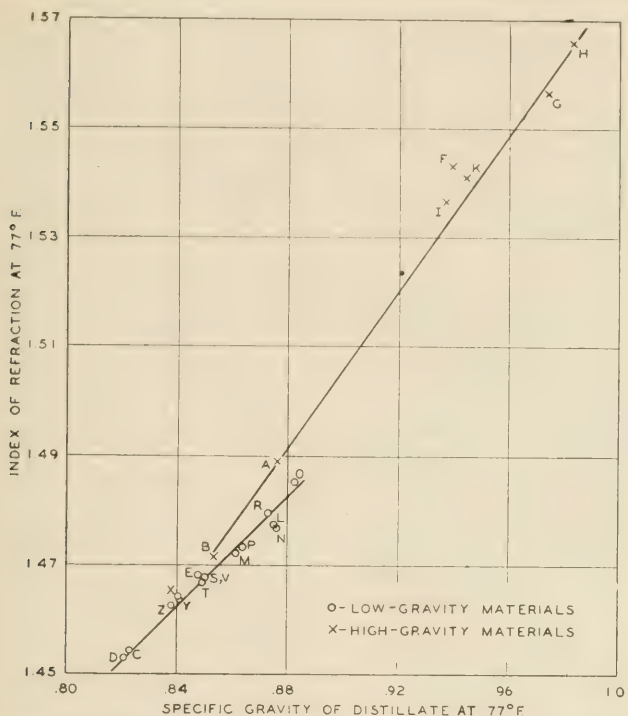


FIGURE 5.—RELATION BETWEEN THE SPECIFIC GRAVITY AND INDEX OF REFRACTION OF THE DISTILLATES.

These products also differ from the others in other respects. Sample D is the only material having a higher percentage of fixed carbon than of asphaltenes in the original material; and the asphaltic residues of both samples C and D have high softening points and penetrations at 32° F.

The test for material insoluble in naphtha and the fixed carbon test are both used as tests of quality and as identification tests. Results of these tests, together with the solubilities in carbon disulphide and carbon tetrachloride, will be discussed in connection with the exposure and laboratory residues.

EXPOSURE OF THIN FILMS OF MATERIAL DESCRIBED

To determine how closely the laboratory evaporation tests indicate the amount of volatile matter lost under actual conditions of exposure and the consistency and character of the resulting residue, relatively thin layers of the materials under investigation were exposed to the action of the sun and air. These samples were not exposed in a manner directly comparable to service conditions, but it is believed that the thicker layers used in this work were not acted upon more severely by the elements than are the relatively thin films of asphaltic materials coating the aggregate in exposed road surfaces. The exposures were made in boxes of the same type as those used in the work of Hubbard and

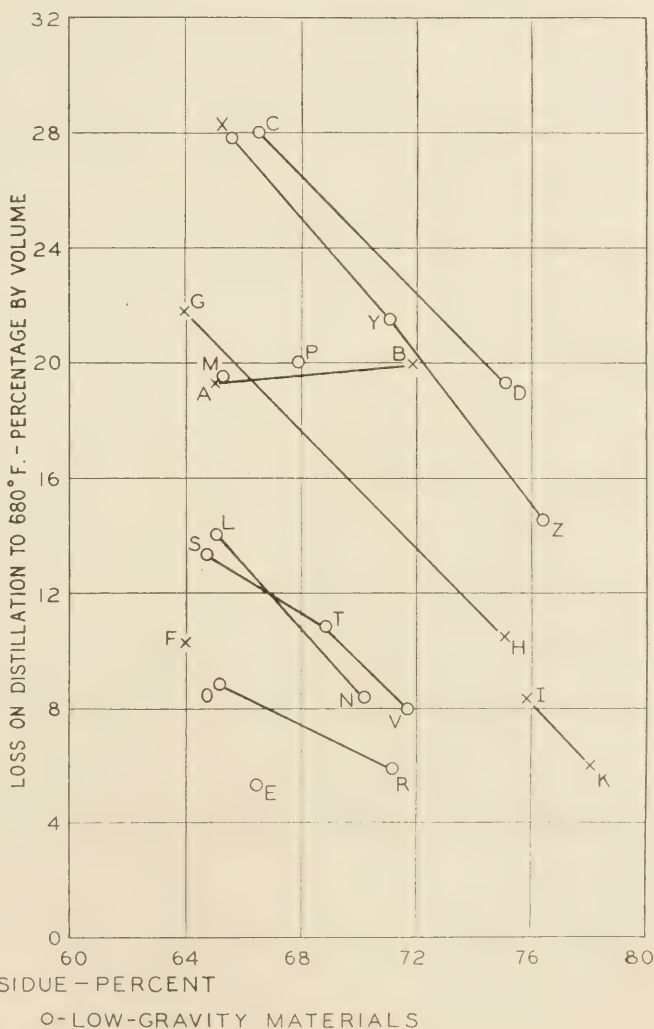
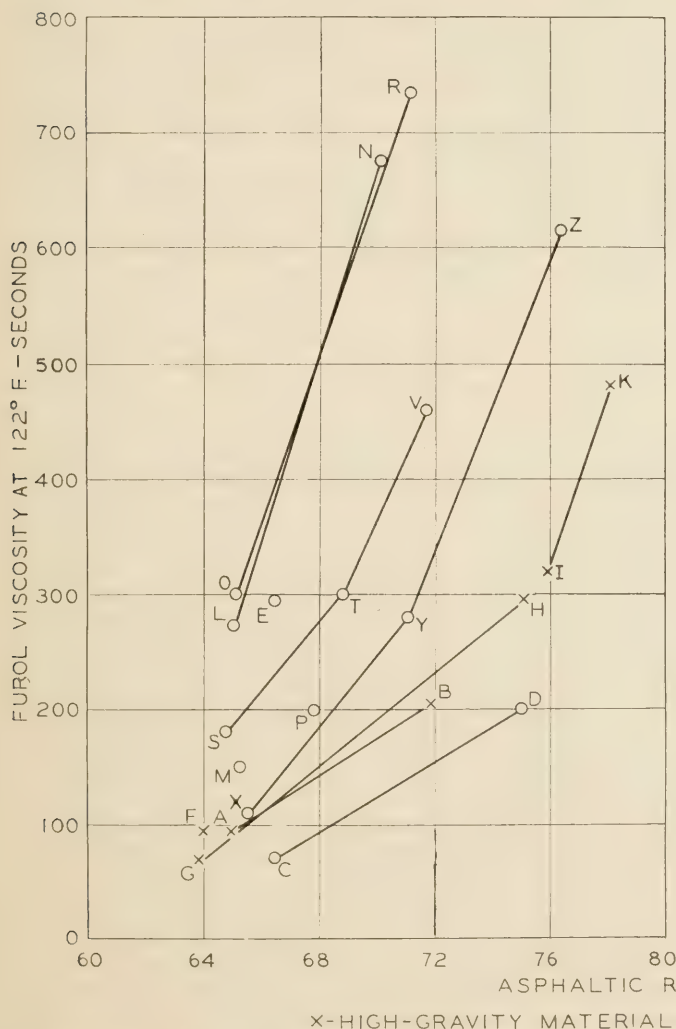


FIGURE 6.—RELATION BETWEEN THE PERCENTAGE OF ASPHALTIC RESIDUE AND THE FURUL VISCOSITY AT 122° F. AND THE PERCENTAGE OF DISTILLATE BY VOLUME.

Reeve,<sup>2</sup> Reeve and Anderton,<sup>3</sup> and Reeve and Lewis.<sup>4</sup> A sketch of the box is shown in figure 7.

These boxes were made of  $\frac{3}{4}$ -inch wood and had interior dimensions of 30 by 24 by 3 inches. A plate glass cover resting on strips of felt fastened to the edges of each box made a tight joint and excluded all dust and dirt. A current of air, which had been passed through a wash bottle containing sulphuric acid to remove the dust and eliminate the moisture, was admitted through the bottom of the box and escaped through slots in the side serving to carry off the vapors formed. The slots were protected from the rain by thin boards extending from the top of the box at an angle of about  $45^\circ$ . Cotton batting inserted in the slots excluded dust from the outside air. A thermometer in each box provided a means of determining the temperature which was recorded hourly from 9 a.m. to 4 p.m.

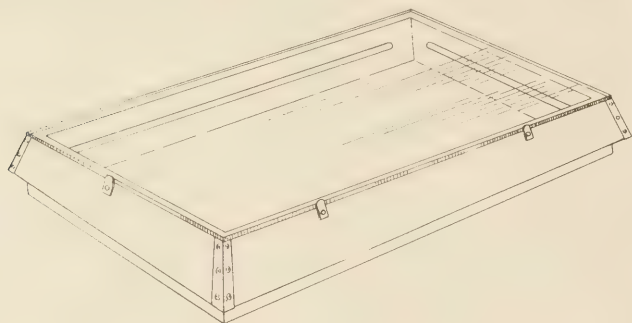


FIGURE 7.—BOX USED IN EXPOSURE TESTS.

The use of plate glass instead of quartz as cover eliminated some actinic rays. This and the absence of moisture probably makes these exposures not fully comparable to outdoor exposure. However, the work of O. G. Streiter<sup>5</sup> shows that a variety of asphalts exhibited the same type of changes when exposed in an accelerated weathering tester as when outdoors under actual weather conditions. It is thought that the presence of water would not have influenced greatly the change in physical and chemical characteristics found in these exposures.

Two samples of each material were placed in seamless, flat bottom tins having a diameter of  $5\frac{1}{2}$  inches and a depth of  $\frac{5}{8}$  inch. Fifty cubic centimeters of material were used to obtain a uniform film or layer thickness of about one-eighth inch (0.128 inch). The samples were so arranged in the exposure boxes and the boxes so placed that no obstruction prevented the action of sunlight. The assembly of the exposure boxes and distributions of samples at start of exposure are shown in figure 8.

The samples were placed in the boxes June 9, 1932, and during the following 15 weeks the minimum and maximum temperatures recorded on the thermometer in the boxes were  $60^\circ$  and  $208^\circ$  F. (9 a.m. to 4 p.m. period only). The average daily minimum and maximum temperatures were  $105^\circ$  and  $175^\circ$  F. The maximum temperature was usually obtained at noon and the minimum in the morning or evening. As the test was conducted to obtain comparative results, the actual

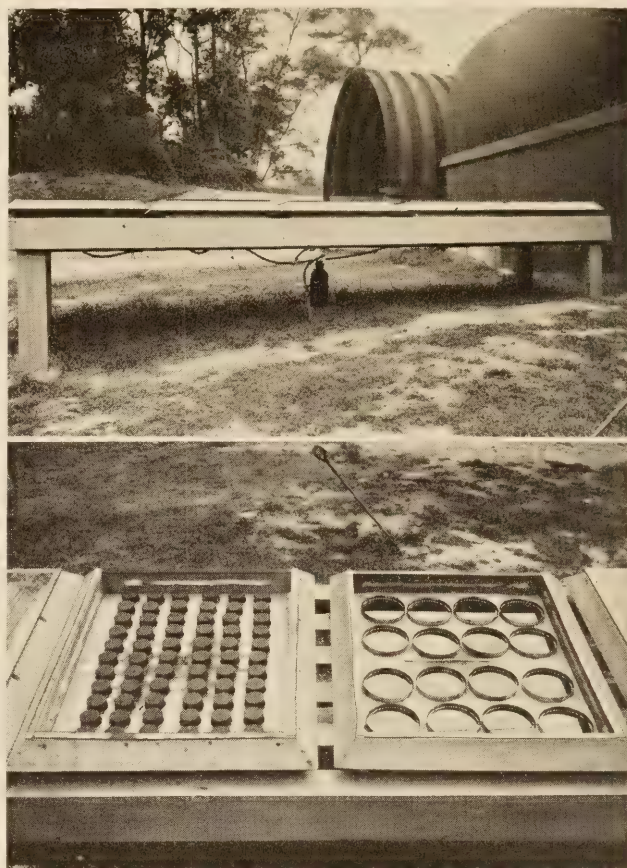


FIGURE 8.—THIN FILMS OF SLOW-CURING MATERIAL AND SPECIMEN CYLINDERS EXPOSED TO SUN.

temperature of each box is not important as long as all the boxes were at the same temperature. Variations in temperature within the several boxes were slight and could not materially affect the results. The samples were weighed at intervals and the percentage of loss calculated. One set was removed for test at the end of 10 weeks and the other at the end of 15 weeks. The percentage of loss at the different stages of the exposures are shown in table 4. The surface conditions of the various materials at the end of 15 weeks exposure are shown in figures 9, 10, 11, and 12.

O. G. Streiter, in studying the progress of the deterioration in asphalts on weathering, resorted to the determination of the oils, resins, and asphaltenes, according to the scheme of Marcusson. The deterioration is shown by a decrease in oils and resins and an increase in asphaltenes. Hubbard and Reeve resorted to consistency tests and tests of solubility in naphtha and carbon disulphide. Reeve and Lewis used these and the softening-point determination and the fixed-carbon test to show the changes which had occurred in terms generally used in routine asphalt testing. The residues after 10 and 15 weeks of exposure were subjected to the tests used by Hubbard and Reeve and Reeve and Lewis, and also to determinations of specific gravity, ductility at two temperatures, and solubility in carbon disulphide and carbon tetrachloride.

In order to compare the residues after exposure in all particulars with the residues developed in the accelerated laboratory tests, the laboratory residues from the 20-gram volatilization test, the distillation

<sup>2</sup> The Effect of Exposure on Bitumens. *Jour. Industrial and Engineering Chemistry*, vol. 5, (1913) 5. A paper presented at Eighth International Congress of Applied Chemistry.

<sup>3</sup> The Effect of Exposure on Tar Products. *Jour. Franklin Institute*, October, 1916.

<sup>4</sup> The Effect of Exposure on Some Fluid Bitumens. *Jour. Industrial and Engineering Chemistry*, vol. 9, no. 8, p. 743.

<sup>5</sup> Accelerated Tests of Asphalts, *Bureau of Standards Journal of Research*, vol. 5, August 1930.



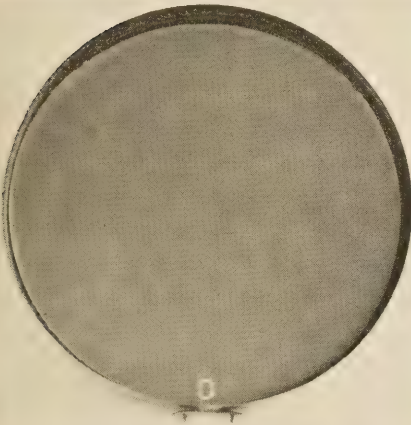
Major portion of the surface is dull, black, and pitted. Surface has shrunk and cracked. Material in bottom of large crack is soft and sticky. Remaining surface is very hard.



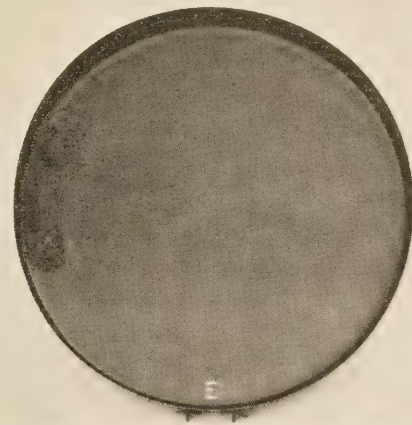
Major portion of the surface is dull, black, and pitted. Surface has shrunk and cracked. Material in bottom of larger cracks is soft and sticky. Remaining surface is very hard.



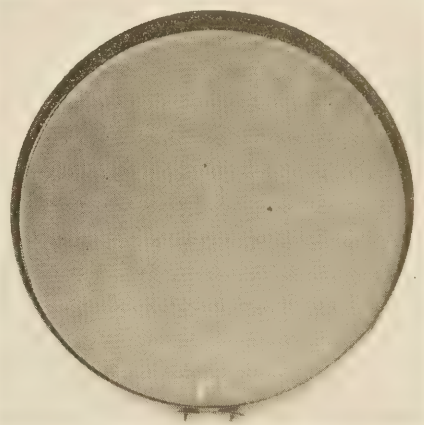
Surface is smooth and glossy to the naked eye but has a mottled appearance when seen under a glass. There is an indication of checking.



Surface is smooth and glossy to the naked eye but has a mottled appearance when seen under a glass. There is an indication of checking.



Surface is dull and mottled except for a small scaly area, caused by material drawing.



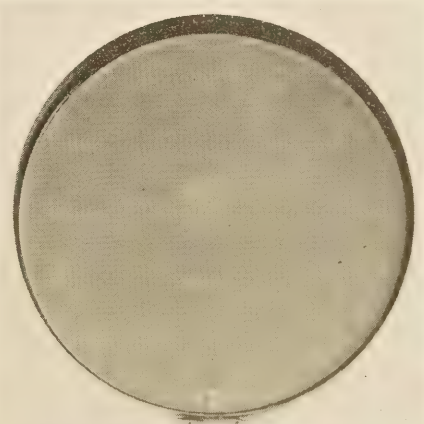
Surface glossy and sticky.



Surface mottled and iridescent; has a greasy feeling.

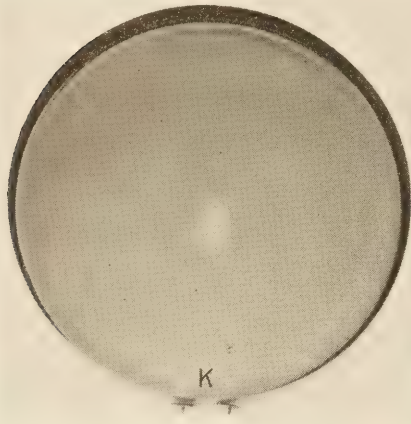


Surface mottled and iridescent; has a greasy feeling.

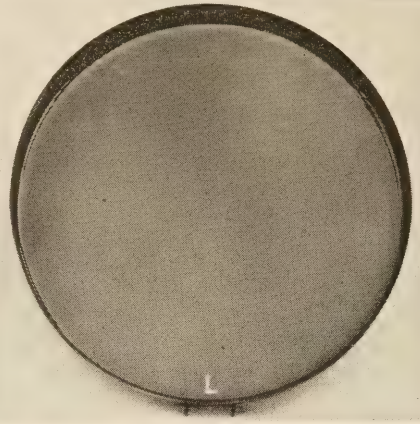


Surface highly iridescent; has a greasy feeling. Has a fine needlelike appearance under a glass.

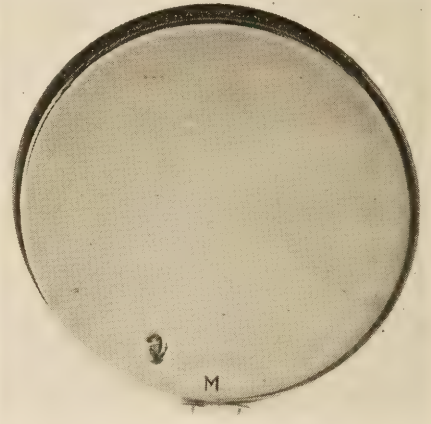
FIGURE 9.—SURFACE CONDITION OF EXPOSED FILMS AT END OF 15 WEEKS.



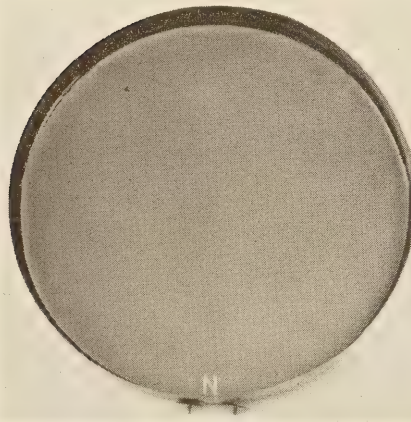
Surface highly iridescent; has a greasy feeling. Has a fine needlelike appearance under a glass.



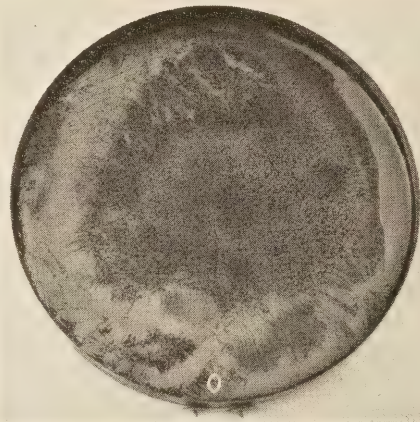
Surface smooth and glossy to the naked eye, but appears drawn when seen under a glass. Surface sticky.



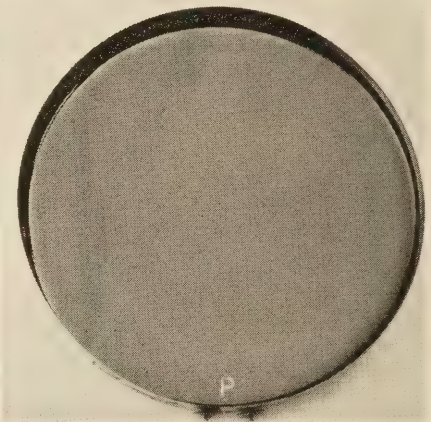
Surface smooth and glossy to the naked eye, but appears drawn when seen under a glass. Surface sticky.



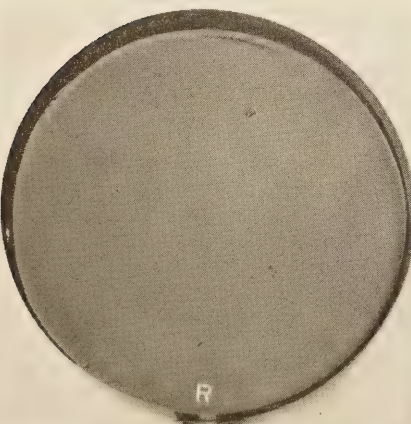
Surface smooth and glossy to the naked eye, but appears drawn when seen under a glass. Surface sticky.



Surface rough and pitted, and black and glossy. Surface around outer edge shows few smooth glossy spots. There is extreme drawing in some areas.



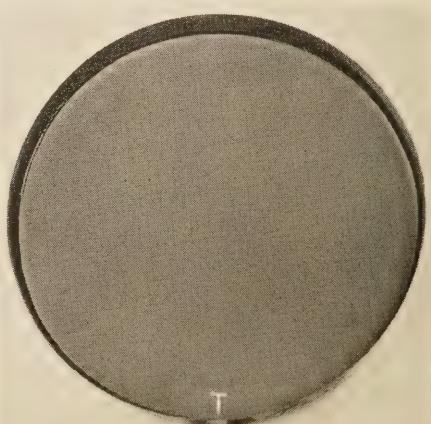
Surface smooth and glossy to the naked eye, but appears drawn when seen under a glass. Surface sticky.



Surface smooth and glossy to the naked eye, but appears drawn when seen under a glass. Surface sticky.

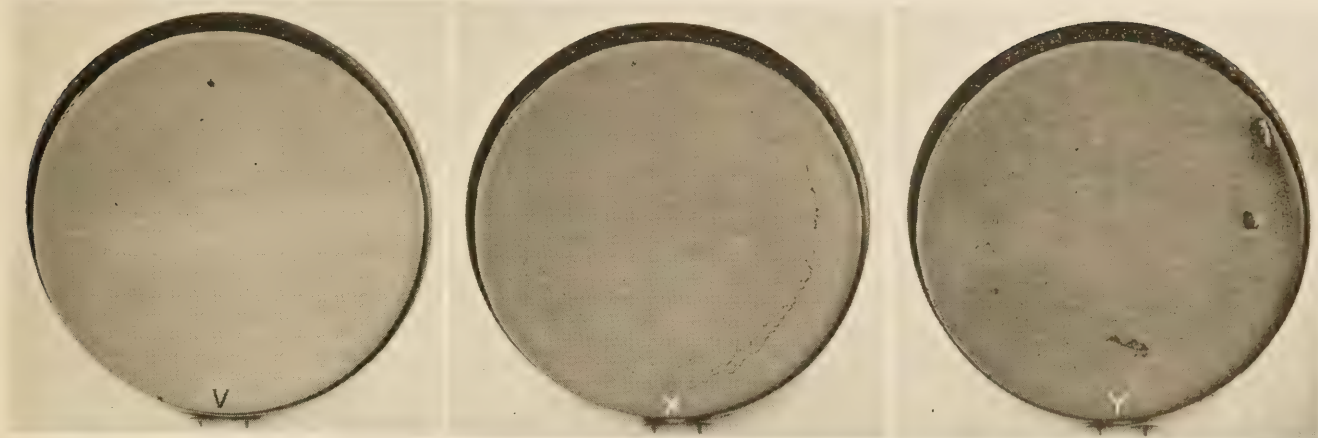


Surface smooth and glossy; appears slightly drawn when seen under a glass.



Surface smooth and glossy; appears slightly drawn when seen under a glass.

FIGURE 10.—SURFACE CONDITION OF EXPOSED FILMS AT END OF 15 WEEKS.

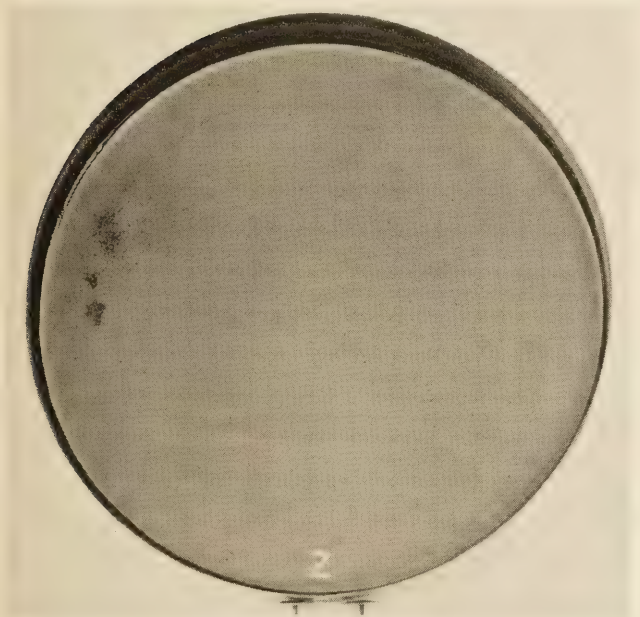


Surface smooth, glossy, and sticky.

Surface glossy, with a network of checking over most of surface.

Surface smooth and glossy, with a network of checking over most of surface.

FIGURE 11.—SURFACE CONDITION OF EXPOSED FILMS AT END OF 15 WEEKS.



Surface glossy and slightly drawn. Some checking over most of surface.

FIGURE 12.—SURFACE CONDITION OF EXPOSED FILM AT END OF 15 WEEKS.

test and the asphaltic-residue test were subjected to the same tests made on the residues after exposure. The results of the tests on both the laboratory residues and residues after exposure are given in table 5.

**LOSSES IN DISTILLATION TEST MOST NEARLY APPROXIMATE LOSSES RESULTING FROM EXPOSURE**

An examination of table 4 shows that all of the materials lose in weight rather rapidly at first and more slowly as the time of exposure increases. There is, however, a great deal of difference in the rate of loss. If the loss in weight at the end of the 15-week period is considered as the maximum possible loss under conditions of this test, then in 2 days the exposure samples lose from 13 percent (sample H) to 61 percent (sample C) of their volatile matter; in 12 days they lose from 21 percent (sample G) to 74 percent (sample

TABLE 4.—Rate of loss in thin film exposure

Identification	Percentage loss of bitumen					
	Time of exposure days					
	2	12	35	50	70	105
A	4.2	6.9	14.4	15.5	16.9	18.2
B	6.5	9.4	16.0	17.4	18.9	19.8
C	16.4	20.0	25.1	25.6	26.3	26.9
D	10.0	12.9	17.8	18.2	18.6	18.7
E	2.7	3.5	6.7	7.3	8.6	8.3
F	5.3	4.8	11.5	12.7	14.4	16.0
G	3.6	5.0	16.2	18.9	22.1	23.7
H	2.1	4.0	11.7	13.3	15.0	16.3
I	2.8	4.0	9.9	11.3	13.1	13.7
K	2.3	3.4	8.1	9.4	10.9	11.5
L	4.3	6.7	13.5	14.6	15.7	17.7
M	9.9	11.5	18.4	19.1	20.2	20.4
N	2.5	3.7	9.3	10.4	11.3	11.9
O	2.6	3.9	9.2	10.2	11.4	11.5
P	7.3	11.0	18.5	19.9	22.4	22.6
R	2.0	2.6	7.1	8.1	9.3	9.5
S	3.4	5.0	12.3	13.5	14.8	17.1
T	2.5	3.8	10.3	11.6	13.2	13.7
V	4.1	5.0	9.1	10.3	11.5	11.8
X	8.4	13.8	24.1	25.6	27.4	27.9
Y	6.6	10.1	19.3	20.5	21.6	22.5
Z	3.3	5.9	14.1	15.2	16.5	17.0

C); and in 35 days from 68 percent (sample G) to 95 percent (sample D). Samples C, D, and M lost 48 percent or more of their volatile matter in 2 days while none of the other samples lost more than 35 percent. These three samples likewise lost a greater percentage of their volatile matter in 12 and 35 days than the other materials. At the end of 10 weeks all of the samples had lost at least 87 percent of their volatile matter and all but 6 had lost over 95 percent. It is reasonable to suppose that the losses at the end of a 15-week period would not have been increased materially had the exposure continued for a longer time.

Figure 13 is a comparison of the 15 weeks' exposure losses and the losses occurring in the laboratory control tests. The percentage of loss in the 15 weeks' exposure is about the same as the total loss in the distillation test, about 1½ times as great as the loss in the 20-gram volatilization test and twice as great as the loss in the 50-gram volatilization test. When the exposure losses are compared with the losses in the asphaltic residue test, however, no reasonably consistent relation is found. The losses in the 15-week exposure samples vary from one-fourth of the loss occurring in the asphal-

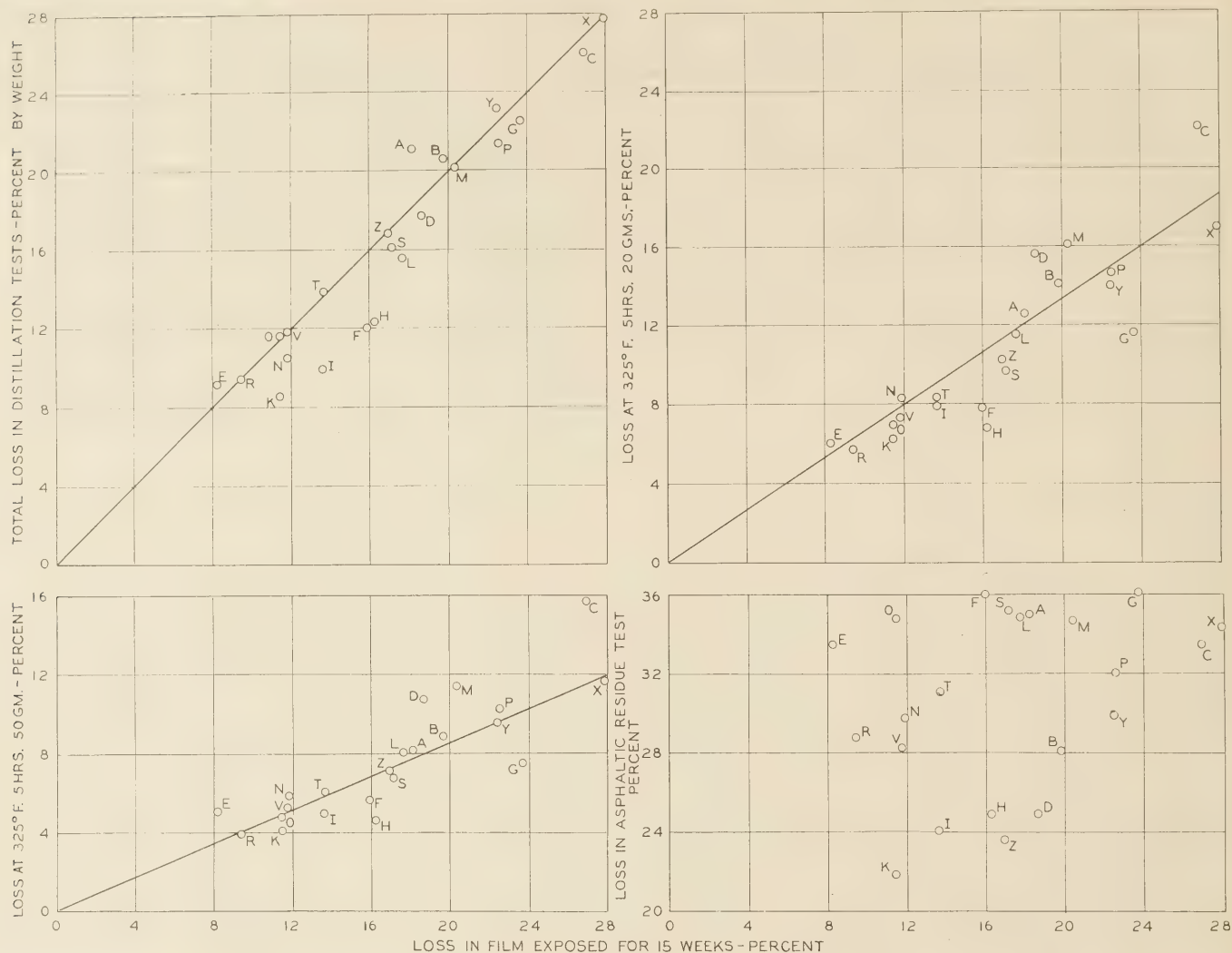


FIGURE 13.—COMPARISON OF THE PERCENTAGE OF LOSS AFTER 15 WEEKS OF EXPOSURE WITH LOSS IN THE LABORATORY EVAPORATION TESTS.

tic residue test for sample E to four-fifths of the loss occurring in the asphaltic residue test on sample X, with an average for the entire group of slightly over one-half of the loss which takes place when these liquid asphaltic materials are reduced to asphaltic residues of 100 penetration by the usual laboratory methods.

The comparisons show that the total loss by weight in the distillation test (including loss on cooling) more nearly approximates the ultimate loss which occurs when these materials are exposed under the conditions of this test than do the losses which occur in the volatilization or asphaltic residue tests.

The losses in the various laboratory and exposure tests are of importance when comparisons are made of the consistencies of the residues as given in table 5. Although the losses in the distillation test and the 15-week exposure test are about equal the distillation residues are fluid and the exposure residues are semi-solid. In spite of the fact that the losses in the exposure test are considerably less than the losses in the asphaltic residue test, the exposure residues are in all but two cases (samples D and E) harder than the corresponding asphaltic residues and in those cases the exposure residues were comparable to an asphaltic residue of 100 penetration.

MATERIALS HIGHLY ALTERED BY EXPOSURE

Bituminous materials upon exposure undergo changes which are due to something more than mere loss of volatile matter. This has been discussed in the papers referred to on page 92 and is demonstrated by these tests. Reeve and Lewis showed that, for equivalent losses, atmospheric exposed samples developed harder consistencies than 20-gram volatilization samples. In the present investigation the exposed samples produced residues much harder than the distillation residues and, in all except two cases, harder than the asphaltic residues although the loss in the exposure test was about the same as the loss in the distillation test and much less than the loss in the asphaltic residue test.

A study of the characteristics of the residues after exposure and the asphaltic residues produced under laboratory conditions is of interest. It has been demonstrated that when asphaltic materials are subjected to high temperatures such as are reached in the asphaltic residue test, they are highly susceptible to oxidation, producing chemical reactions which result in artificial hardening of the material. Bateman and Delp<sup>6</sup> showed that when Mexican asphalt is sprayed on hot

<sup>6</sup> The Recovery and Examination of the Asphalt in Asphaltic Mixtures, Proc. A. S. T. M., vol. 27, pt. 2, p. 465.





TABLE 6.—Comparison of residues of approximately 100 penetration obtained from the asphaltic residue test and from exposed films of material

Identification	Asphaltic residue				Exposure residue				Time of exposure— weeks			
	Penetration		Softening point ° F.	Ductility	Penetration		Softening point ° F.	Ductility				
	At 77° F.	At 32° F.			At 77° F.	At 32° F.						
A	87	19	4.6	113 110+	0	115	34	3.4	149	3	0.5	10
C	95	25	3.3	118 110+	4.8	117	38	3.1	111	72	4.5	10
D	88	27	3.3	120 110+	4.8	93	29	3.2	113	86	4.5	15
E	104	30	3.5	114 100+	6.0	124	52	2.4	117	23	3.3	15
F	101	9	11.2	108 110+	0	79	12	6.6	109	100	0	15
G	82	8	10.2	109 110+	0	119	23	5.2	103	110+	0	10
H	92	8	11.9	109 110+	0	76	13	5.9	110	110+	0	15
L	93	20	4.7	111 110+	0	87	25	3.5	113	110+	0	10
M	112	19	5.9	110 110+	0	111	31	3.7	109	110+	0	10
N	105	19	5.5	109 110+	0	91	25	3.7	113	110+	0	10
P	107	18	6.0	110 110+	1.0	85	23	3.7	113	110+	0	10
R	102	25	4.1	113 110+	5.5	86	23	3.1	113	102	3.5	10
S	106	20	5.3	113 110+	5.8	83	31	2.7	120	42	3.5	15
T	102	21	4.9	114 110+	0.7	95	30	3.2	115	60	3.2	10
V	99	21	4.7	113 110+	4.5	105	29	3.6	112	81	3.8	10

lowered and, in the case of sample A, an extremely non-ductile residue has been produced. Materials which are highly ductile at 77° F. generally have little if any ductility at low temperatures and in the case of samples L, M, and N, the fact that the residues after exposure have a higher ductility at 34-35° F. than the corresponding asphaltic residues may be due to the fact that the residues after exposure may actually have a lesser ductility at 77° F. than the asphaltic residues. This difference, if it exists, is not shown by the test results because of the limited capacity of the ductility machine available.

The softening point of the residues after exposure of samples B and O, although not shown in table 6 because of their low penetrations at the end of 10 weeks, are also abnormally high when compared with other residues having practically the same consistency. The ductility of the residues after exposure of these two samples is also exceedingly low. It will be shown later by other tests that there are changes in characteristics in the asphaltic residues which may be attributed to oxidation. Nevertheless, from a study of these data it is apparent that the effects of oxidation at comparatively low temperatures over a relatively long period of time are more pronounced than at high temperatures for a comparatively short period.

Attention is called to the behavior of samples A, B, and O. It has been held by some authorities that liquid asphaltic materials which give ductile residues are desirable materials for road use and that products giving non-ductile residues are unsuitable for many types of road construction. While producing a residue of satisfactory properties in the asphaltic residue test, samples A, B, and O, on exposure, lost practically all ductility and were materially altered as shown by their photographs in figures 9 and 10. From the standpoint of surface appearance these samples are the worst of the group.

CONSIDERABLE FIXED CARBON AND MATERIAL INSOLUBLE IN NAPHTHA FORMED IN ALL MATERIALS DURING EXPOSURE

Since both the percentage of material insoluble in naphtha and the percentage of fixed carbon are considered to be characteristic of the source and method of manufacture of different types of asphaltic materials, the relations between these two values for the original materials and all of their residues are plotted in figure 14. Only a very general relation is indicated for the

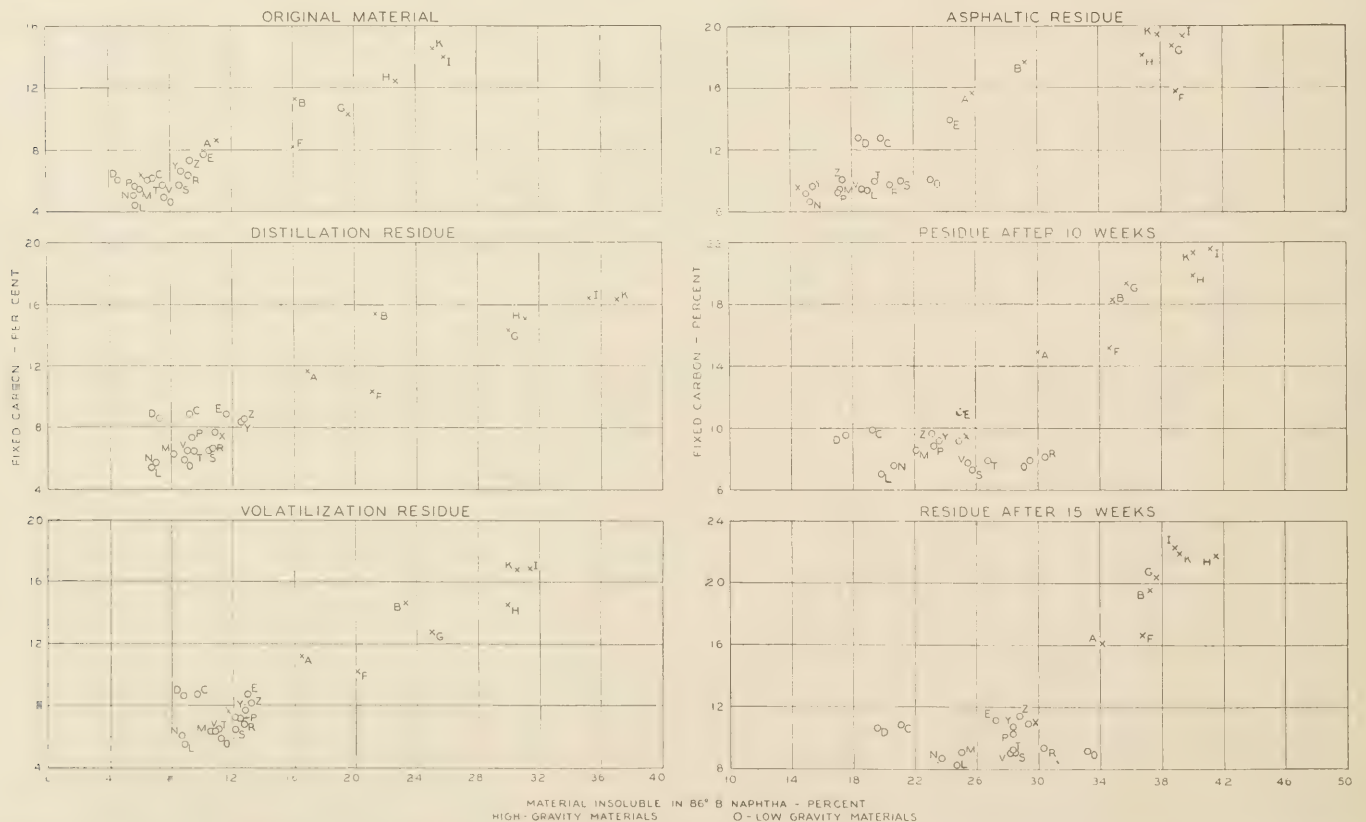


FIGURE 14.—RELATION BETWEEN THE PERCENTAGE OF FIXED CARBON AND MATERIAL INSOLUBLE IN NAPHTHA IN THE ORIGINAL MATERIALS AND THEIR RESIDUES RESULTING FROM LABORATORY TREATMENT AND FIELD EXPOSURE.

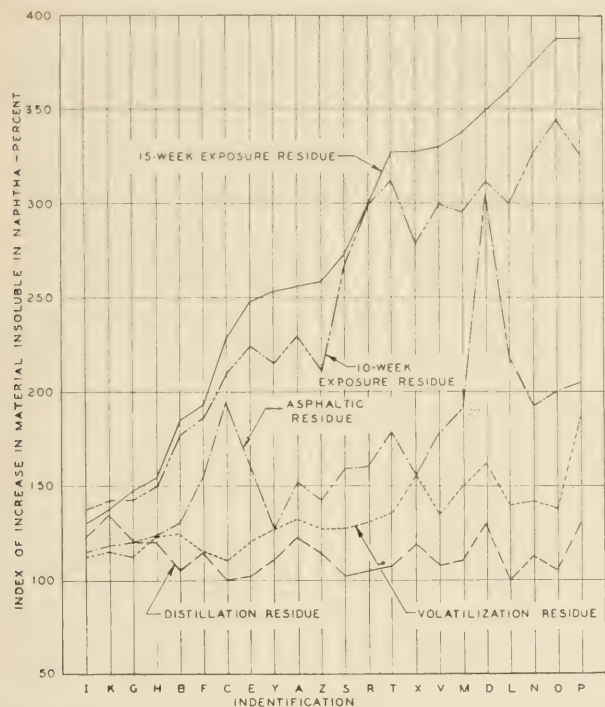


FIGURE 15.—INDEX OF INCREASE IN MATERIAL INSOLUBLE IN NAPHTHA IN THE VARIOUS RESIDUES.

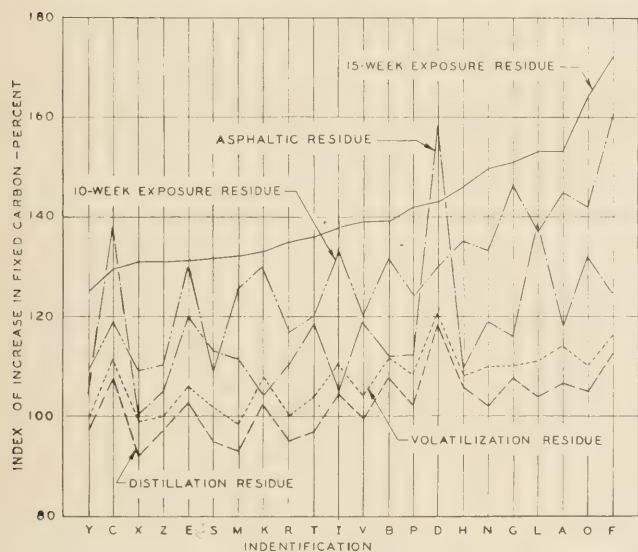


FIGURE 16.—INDEX OF INCREASE IN FIXED CARBON IN THE VARIOUS RESIDUES.

group as a whole. However, in all cases the high-gravity materials have higher percentages of fixed carbon and material insoluble in naphtha than the low-gravity materials. It is of interest to note that materials from the same refinery, and presumably from the same source, continue to show the same relations in the residues as in the original material (samples C, D, S, T, and V).

Anderton<sup>7</sup> found that in the steam distillation of petroleum from various fields the fixed carbon of a residue is dependent only upon the character of the crude material and is unaffected by the distillation. Within the limits of accuracy of the test, the fixed carbon value for any residue can therefore be readily

calculated by knowing the fixed carbon of the original material and the percentage of distillate driven off during the steam distillation. He also found that the percentage of asphaltenes increased in the various residues obtained in fractional steam distillation in greater proportion than could be attributed to loss of volatile matter and concluded that there were changes occurring which resulted in the actual formation of bodies of an asphaltic character.

This later work of Anderton justified to a great extent the use of the fixed carbon and naphtha-solubility tests by Reeve and Lewis in studying the effects of exposure on asphaltic materials. The latter investigator demonstrated from abnormal increases in fixed carbon and naphtha insolubility in samples exposed to oven and atmospheric conditions that polymerization and intermolecular reactions caused by heat, and possibly increased also by the action of light, are very largely responsible for such changes, in addition to those accounted for by oxidation and mere loss of volatile matter. It is concluded therefore that the fixed-carbon and material insoluble in naphtha determinations are of value in a comparison of the laboratory residues and residues after exposure of the liquid asphaltic materials used in this investigation.

If there is no material insoluble in naphtha in the volatile matter and none is formed during the progress of the test, the fraction of material insoluble in naphtha should be equal to the percentage in the original material divided by the percentage of residue. If the residue contains more than this calculated or theoretical amount of material insoluble in naphtha it must have been formed during the test. Therefore the actual percentage of naphtha-insoluble matter in the residue divided by the calculated percentage may be called the ratio of increase in material insoluble in naphtha. This ratio multiplied by 100 is called the index of increase and is used to indicate the relative changes which have taken place in the various residues. The relative changes in the fixed carbon content of the various residues may also be determined in the same manner.

In figures 15 and 16 the indexes of increase in naphtha-insoluble material and fixed carbon in the residues, are plotted for each sample and each method of test. When the actual amount of fixed carbon or material insoluble in naphtha is equal to the theoretical or calculated amount, the index of increase, as shown in figures 15 and 16, is 100. It will be noticed that in all cases the increase in naphtha-insoluble material is much greater than the increase in fixed carbon, the index for the residues after 15 weeks' exposure ranging from about 125 to 375 for the material insoluble in naphtha as compared with indexes of 125 to 175 for fixed carbon.

The change in fixed carbon and naphtha-soluble material during the distillation test is slight, the index varying from 100 to 135 for naphtha-insoluble material and from 92 to 117 for fixed carbon. It is probable that there was no actual decrease in fixed carbon, but slight inaccuracies in the test doubtless had a noticeable effect in the distillation residues where the changes were small. The increase in fixed carbon in the volatilization residue is always higher than in the distillation residue, and the same is true for the material insoluble in naphtha except in three cases, but the curves are almost parallel, especially in the case of fixed carbon. In the case of the asphaltic residue the indexes are high when the time of reduction is long, as in the case of samples C and D. In these cases the asphaltic residue

<sup>7</sup> B. A. Anderton. Some Relations Between the Characteristics of Steam-Distilled Petroleum Residuals. Vol. 20, pt. 11, Proc. A.S.T.M. 1920.

has a higher index for fixed carbon than has the residue resulting from a 15-week exposure and the indexes for increase in naphtha-insoluble material approach the values for the residues after 10 weeks' exposure. The indexes are low when the time of reduction is low, as in samples X and Y, where the increase in material insoluble in naphtha is no greater than in the volatilization residue. It is also interesting to note that in general the residues of the high-gravity materials have a lower index for material insoluble in naphtha than have the low-gravity materials. In the case of the index for fixed carbon, however, the high-gravity materials do not fall into any one group.

It will be seen that the changes in material insoluble in naphtha and in the fixed carbon content are greatest in the residues after 15 weeks' exposure; they are next greatest in residues after 10 weeks' exposure, then in the asphaltic residues, then in the volatilization residues, and least in the distillation residues. The data given clearly demonstrate that all the accelerated evaporation tests now in use for the control of asphaltic materials produce residues which have been less altered by artificial hardening and oxidation than are residues resulting from exposure to the sun and to atmospheric conditions. This corroborates the work of Reeve and Lewis.

#### FREE CARBON AND CARBENES FORMED ONLY IN HIGH-GRAVITY MATERIALS DURING EXPOSURE

By definition, bitumen is the material soluble in carbon disulphide, asphaltenes are that portion of bitumen insoluble in 86° B. naphtha, and carbenes are that portion of bitumen insoluble in carbon tetrachloride. Referring to tables 3 and 5 it will be seen that the percentage of organic matter insoluble in carbon tetrachloride is exceedingly small in the original materials having low specific gravities; and that there is no great increase in the material insoluble in carbon tetrachloride in their various residues. In these low-gravity materials, therefore, a solubility in carbon tetrachloride is practically the same as a solubility in carbon disulphide; and the material insoluble in 86° B. naphtha may be considered as essentially asphaltenes.

The high-gravity products, however, which originally had from 0.50 percent in the case of sample F to 2.88 percent in the case of sample I of material insoluble in carbon tetrachloride, developed residues which showed substantial increases in material insoluble in carbon tetrachloride. It was indicated clearly, in the case of these high-gravity materials, that solubility in carbon tetrachloride was by no means the same as solubility in carbon disulphide; and it was therefore necessary to test these high-gravity materials and their residues for solubility in carbon disulphide. As shown in tables 3 and 5, these materials have appreciable amounts of material insoluble in carbon disulphide, in their original condition, and in their various residues. In the case of these high-gravity products, therefore, the organic matter insoluble in carbon tetrachloride, as reported in these tables, does not represent carbenes and the organic matter insoluble in 86° B. naphtha does not represent asphaltenes (since they are based on total material rather than total bitumen).

In table 7 the data on solubility have been tabulated to show not only the insoluble matter in the high-gravity materials and their residues but what, by definition, are free carbon (insoluble in carbon disulphide), carbenes, and asphaltenes. The greatest differences between percentage of organic matter insoluble in 86°

B. naphtha and percentage of asphaltenes are in the residues after exposure where the free carbon and carbenes have increased to a great extent. In four cases the free carbon and carbenes are greater in the residue from the volatilization test than in the asphaltic residue, the effect of time in the oven test probably being more pronounced than the higher temperature used for a short period in the asphaltic residue test. In two cases the carbenes are less in the distillation residue than in the original material. In figure 17 the volatile matter and the insoluble matter in 86° B. naphtha, carbon tetrachloride, and carbon disulphide are shown for the original materials and their various residues. All percentages are expressed in terms of the weight of the original material.

TABLE 7.—Comparative solubilities of the high-gravity materials and their residues

Identification	Solubility test	Original material	Distillation residue	20-gram volatilization residue	Asphaltic residue	10-week exposure residue	15-week exposure residue
		Percent	Percent	Percent	Percent	Percent	Percent
A	CS <sub>2</sub> insoluble	0.19	0.24	0.39	0.39	0.87	1.61
	CCl <sub>4</sub> insoluble	.83	.47	1.33	1.46	3.49	4.25
	Naphtha insoluble	10.95	16.89	16.50	25.84	29.95	34.06
	Carbenes	.64	.23	.94	1.07	2.64	3.68
	Asphaltenes	10.78	16.69	16.17	25.55	29.34	32.98
B	CS <sub>2</sub> insoluble	.17	.38	.28	.60	1.06	2.50
	CCl <sub>4</sub> insoluble	2.17	1.83	4.88	3.41	8.27	9.11
	Naphtha insoluble	16.07	21.31	23.26	29.24	34.76	37.16
	Carbenes	2.00	1.45	4.61	2.83	7.29	6.78
	Asphaltenes	15.93	21.01	23.04	28.81	34.06	35.55
F	CS <sub>2</sub> insoluble	.07	.20	.10	.75	2.54	3.69
	CCl <sub>4</sub> insoluble	.50	1.06	1.73	3.12	8.15	9.59
	Naphtha insoluble	16.01	21.08	20.08	38.87	34.62	36.68
	Carbenes	.43	.86	1.63	2.38	5.76	6.13
	Asphaltenes	15.95	20.92	20.00	38.41	32.92	34.25
G	CS <sub>2</sub> insoluble	.19	.29	.95	.71	5.10	6.56
	CCl <sub>4</sub> insoluble	1.58	2.84	4.95	4.98	12.22	14.79
	Naphtha insoluble	19.56	30.00	25.03	38.70	35.65	37.58
	Carbenes	1.39	2.56	4.04	4.30	7.50	8.81
	Asphaltenes	19.41	29.80	24.31	38.26	32.19	33.20
H	CS <sub>2</sub> insoluble	.58	.49	1.84	1.00	4.14	6.41
	CCl <sub>4</sub> insoluble	2.03	3.02	5.68	4.02	11.78	13.40
	Naphtha insoluble	22.74	31.09	29.91	36.81	40.04	41.51
	Carbenes	1.46	2.54	3.91	3.05	7.97	7.47
	Asphaltenes	22.29	30.75	28.60	36.17	37.45	37.50
I	CS <sub>2</sub> insoluble	.08	.08	.69	.24	5.96	6.17
	CCl <sub>4</sub> insoluble	2.88	4.54	7.35	3.84	14.94	15.61
	Naphtha insoluble	25.83	35.21	31.38	39.40	41.08	38.83
	Carbenes	2.80	4.46	6.71	3.61	9.55	10.06
	Asphaltenes	25.77	35.16	30.90	39.25	37.35	34.81
K	CS <sub>2</sub> insoluble	.09	.17	.47	.30	4.51	5.41
	CCl <sub>4</sub> insoluble	2.83	4.71	6.70	5.21	14.66	15.25
	Naphtha insoluble	25.08	37.01	30.48	37.75	40.03	39.12
	Carbenes	2.74	4.55	6.26	4.92	10.63	10.41
	Asphaltenes	25.01	36.90	30.15	37.56	37.20	35.63

Both in figure 17 and table 7 it is apparent that the total material insoluble in naphtha, which in many asphaltic materials may be considered essentially the same as asphaltenes (bitumen insoluble in naphtha), is, in the case of these high-gravity materials and their residues, a combination of free carbon, carbenes, and asphaltenes. The percentage composition of the material insoluble in naphtha, as given in table 8, indicates that free carbon and carbenes are much more pronounced in the residues after exposure than in the residues from the laboratory tests, six of the 15-week residues after exposure having materials insoluble in naphtha which contained about 25 to 40 percent of free carbon and carbenes.

It has been stated by some authorities that free carbon and carbenes are the result of further condensation or polymerization of asphaltenes. Under the conditions of the various laboratory tests and under the weathering conditions of the exposure tests, the 15 low-gravity products did not develop residues with a gain in organic matter insoluble in carbon tetrachloride. The 7 high-gravity products, which had some free

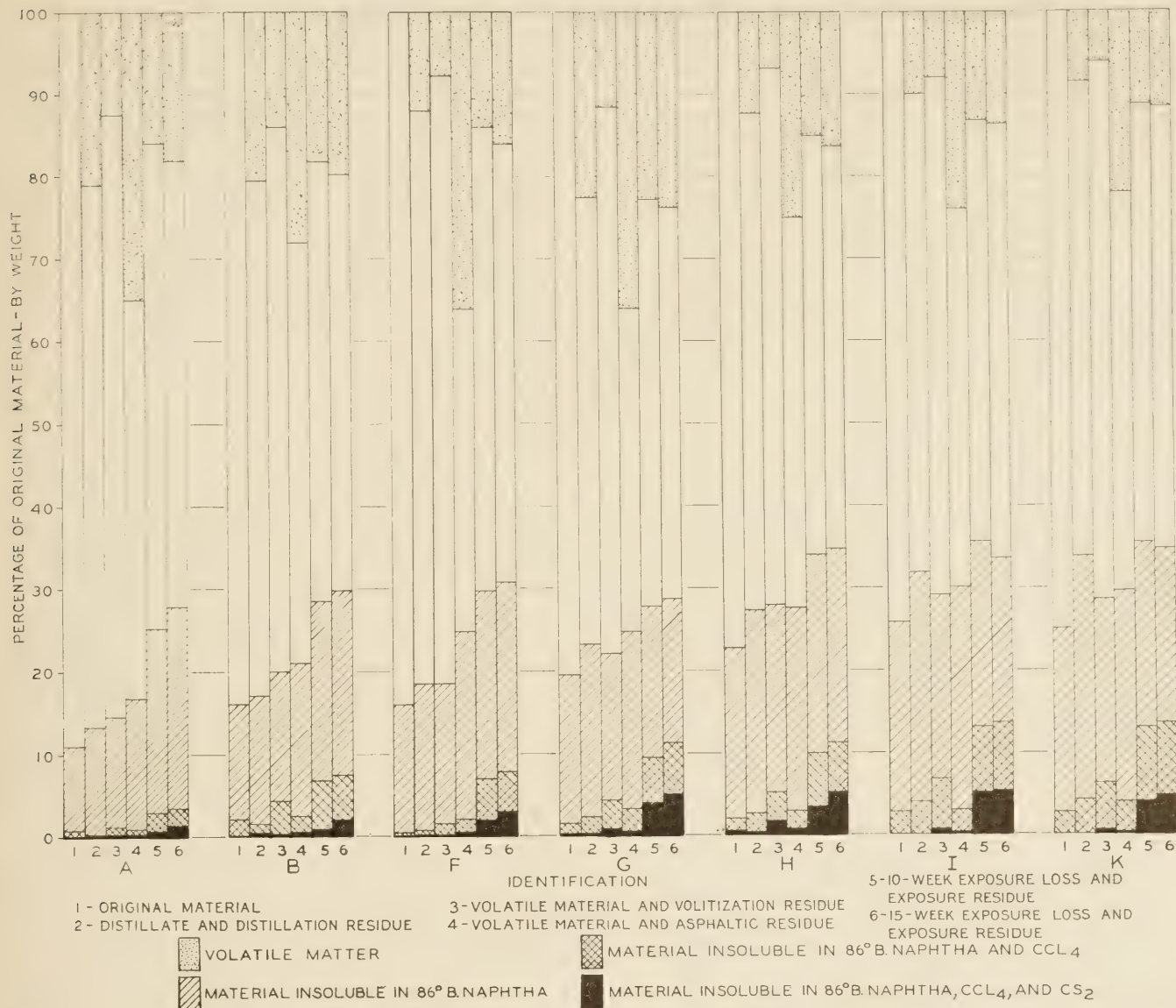


FIGURE 17.—COMPOSITION OF THE HIGH-GRAVITY MATERIALS, LOSS IN VARIOUS TESTS AND COMPOSITION OF THEIR RESIDUES AS DETERMINED BY SOLUBILITY TESTS.

carbon as well as an appreciable amount of carbones, developed considerable percentages of these constituents, especially in the residues after exposure. Evidently carbonization, which takes place when bituminous materials are subjected to high temperature and which progresses more rapidly when they are subjected to sunlight, is intensified when the materials initially have free carbon and carbones present together in appreciable amounts. Therefore, specifications calling for a high solubility in carbon disulphide and carbon tetrachloride should be advantageous in minimizing the possibilities of carbonization playing a part in the weathering of the materials in road service.

**HUBBARD-FIELD STABILITY TEST USED AS AN INDICATION OF THE RELATIVE BONDING STRENGTH OF FLUID ASPHALTIC MATERIALS WHEN COMBINED WITH A STANDARD AGGREGATE**

Slow-curing liquid asphaltic materials are not suitable for surface treatments where exceedingly rapid loss of volatile matter and early hardening are essential to develop high bonding strength. Whether used in mixed surfaces or in penetrative treatments these products have, however, some initial bonding strength and de-

velop additional bonding strength depending upon the design of the surface, climatic conditions, and the inherent qualities of the materials themselves.

In order to evaluate adhesiveness or bonding strength tests were made with the Hubbard-Field stability machine,<sup>8</sup> which has been used to demonstrate the effect of consistency and other characteristics of asphalt cements on the stability or resistance to displacement of sheet-asphalt mixtures.

The first phase of this study, the determination of the bonding strength of the original materials, and of their distillation and asphaltic residues, involved the preparation of 3 sets of cylinders for the Hubbard-Field stability test. In each set there were 3 specimens for each material. In 1 set 16.6 percent by volume of the original materials (6.6 to 7.7 percent by weight depending upon their specific gravity) was mixed with 83.4 percent by volume of the standard sand. In the other 2 sets the same percentage by weight of distillation residue and asphaltic residue was

<sup>8</sup>A study of certain factors affecting the stability of asphalt paving mixtures. Prevost Hubbard and H. C. Field. Proc. A.S.T.M., vol. 26, pt. II, pp. 577-601.

used as was used in the cylinders made with the original materials. This gave an almost constant percentage of bitumen by volume in the cylinders containing distillation residue and asphaltic residue. These mixes were tested immediately for stability at 77° F. and for the purpose of discussion are called series 1 cylinders.

The second phase of the study, the determination of the development of bonding strength, involved the preparation of 6 sets of 3 cylinders each for each material using the same standard aggregate as was used in series 1 and the same percentage of the original materials by volume.

Three sets were placed in a Freas oven regulated at 140° F. and sets were removed at the end of 1, 7, and 14 days. The cylinders were weighed before and after curing and the loss in weight expressed as a percentage of the bituminous material present in the cylinders as made. After weighing, the cylinders were held in an air bath at 77° F. to insure a uniform temperature and then tested at 77° F. for stability.

The other 3 sets were placed in the exposure boxes previously described and subjected to the same exposure conditions as the thin films. Sets were removed at the end of 5, 10, and 15 weeks and tested in the same manner as the oven-cured cylinders. For comparative purposes 2 additional sets of cylinders were made using as a binder the amounts of distillation residue and asphaltic residue which would have been obtained if the bitumen in the cylinders made with the original materials had been subjected to the distillation or asphaltic residue test. These two sets were tested immediately. For the purpose of discussion these are called series 2 cylinders. Table 9 gives the percentage composition of each mix by weight and by volume.

The aggregate was a Potomac River sand which had been separated on standard sieves and recombined for each mix to obtain the following grading:

Passing sieve no.—	Retained on sieve no.—	Percent
10	20	3.9
20	30	6.4
30	40	10.3
40	50	14.2
50	80	30.9
80	100	16.8
100	200	16.7
200		0.8

This sand had a specific gravity of 2.653 and the voids, as determined by the Bureau of Public Roads void determinator,<sup>9</sup> were 34.5 percent. It conforms essentially to Richardson's grading for heavy-traffic sheet asphalt sand without the filler (the material passing the no. 200 sieve), which was omitted not only to provide an open mix for possible loss of volatile matter, but also to eliminate the effect of filler on the bonding strength of the mixtures.

The asphaltic materials and sand were thoroughly mixed by hand. Mixtures containing the original asphaltic materials were mixed at room temperature. Mixtures containing the distillation residues were warmed to 100° F. and those containing the asphaltic residues were heated to 200° F. to expedite mixing and to insure uniform coating. The cylinders were compacted under a load of 3,000 pounds per square inch. The method of compaction recommended by Hubbard and Field was varied in the following particulars:

1. Cylinders containing the original materials and distillation residues were compressed at room temperature.

TABLE 8.—Composition of the total material insoluble in naphtha of the high-gravity materials and their residues

Identification	Naphtha-insoluble matter composed of—	Original material	Distillation residue	20-gram volatilization residue	Asphaltic residue	10-week exposure residue	15-week exposure residue
		Percent	Percent	Percent	Percent	Percent	Percent
A	Free carbon	1.7	1.4	2.4	1.5	2.9	4.7
	Carbenes	5.8	1.4	5.7	4.1	8.7	7.8
	Asphaltenes minus carbenes	92.5	97.2	91.9	94.4	88.4	87.5
B	Free carbon	1.1	1.8	1.2	2.1	3.1	6.7
	Carbenes	12.5	6.8	19.8	9.6	20.8	17.8
	Asphaltenes minus carbenes	86.4	91.4	79.0	88.3	76.1	75.5
F	Free carbon	.4	.9	.5	1.9	7.3	10.1
	Carbenes	2.7	4.1	8.2	6.1	16.2	16.6
	Asphaltenes minus carbenes	96.9	95.0	91.3	92.0	76.5	73.3
G	Free carbon	1.0	1.0	3.8	1.8	14.3	17.5
	Carbenes	7.1	8.5	16.0	11.0	20.0	21.9
	Asphaltenes minus carbenes	91.9	90.5	80.2	87.2	65.7	60.6
H	Free carbon	2.5	1.6	6.2	2.7	10.4	15.4
	Carbenes	6.4	8.1	12.9	8.2	19.1	16.9
	Asphaltenes minus carbenes	91.1	90.3	80.9	89.1	70.5	67.7
I	Free carbon	.3	.2	2.2	.6	14.5	15.9
	Carbenes	10.9	12.7	21.2	9.2	21.9	24.3
	Asphaltenes minus carbenes	88.8	87.1	76.6	90.2	63.6	59.8
K	Free carbon	.4	.5	1.7	.8	11.3	13.8
	Carbenes	10.9	12.3	22.5	13.0	25.4	25.2
	Asphaltenes minus carbenes	88.7	87.2	75.8	86.2	63.3	61.0

TABLE 9.—Composition of cylinders for Hubbard-Field stability test

Identification	Specific gravity of asphaltic material at 77° F.	Percentage of bitumen by weight in all series 1 cylinders and those of series 2 made with original material	Percentage of bitumen by volume in series 1 distillation residue cylinders	Percentage of bitumen by volume in asphaltic residue cylinders of series 1	Series 2—distillation residue cylinders			Series 2—asphaltic residue cylinders		
					Residue, percentage of original material	Percentage of bitumen by weight	Percentage of bitumen by volume	Residue, percentage of original material	Percentage of bitumen by weight	Percentage of bitumen by volume
A	1.001	7.0	16.1	15.9	78.9	5.6	13.1	65.0	4.7	11.0
B	1.013	7.1	16.0	15.8	79.4	5.8	13.3	71.9	5.2	11.9
C	0.942	6.6	15.9	15.7	74.0	5.0	12.3	66.5	4.5	11.0
D	0.955	6.7	16.1	15.9	82.3	5.6	13.7	75.1	5.1	12.4
E	0.976	6.8	16.2	15.9	90.9	6.3	15.3	66.5	4.7	11.3
F	1.066	7.4	16.3	15.9	88.0	6.6	14.7	64.0	4.9	10.9
G	1.079	7.5	16.1	15.9	77.5	5.9	13.0	63.9	4.9	10.7
H	1.101	7.7	16.4	16.2	87.7	6.8	14.7	75.1	5.9	12.7
I	1.082	7.5	16.4	16.0	90.1	6.8	15.0	75.9	5.8	12.6
K	1.088	7.6	16.5	16.2	91.5	7.0	15.3	78.1	6.0	13.1
L	0.972	6.8	16.3	16.0	84.4	5.8	14.1	65.1	4.6	11.2
M	0.967	6.8	16.2	16.0	79.9	5.5	13.4	63.3	4.5	11.2
N	0.981	6.9	16.5	16.2	89.5	6.2	15.0	70.2	4.9	11.9
O	0.972	6.8	16.4	16.0	88.4	6.1	14.9	65.2	4.6	11.2
P	0.967	6.8	16.2	16.0	78.6	5.4	13.2	67.9	4.7	11.4
R	0.979	6.9	16.6	16.2	90.6	6.3	15.3	71.2	5.0	12.1
S	0.952	6.7	16.3	15.7	83.9	5.7	14.1	64.8	4.4	10.7
T	0.960	6.7	16.1	15.9	86.1	5.9	14.5	68.9	4.7	11.5
V	0.966	6.8	16.4	16.1	88.2	6.0	14.7	71.7	5.0	12.2
X	0.951	6.7	16.0	15.8	72.3	4.9	12.0	65.6	4.5	11.0
Y	0.963	6.8	16.1	15.9	76.8	5.3	12.8	71.1	4.9	11.8
Z	0.972	6.8	16.0	16.2	83.2	5.7	13.8	76.4	5.3	12.7

2. Cylinders containing asphaltic residues were compressed at a temperature of 200° F.

3. Cylinders were formed without tamping.

4. In forming cylinders containing original material, pressure was released as soon as the pressure required was reached. With cylinders containing distillation residues and asphaltic residues pressure was maintained for 1 and 2 minutes respectively. In forming cylinders containing asphaltic residues the mold was flooded with water to cool to room temperature as soon as maximum pressure was reached.

Determinations made on the compacted specimens showed the cylinders containing original material to have an average of 34.3 percent voids in the mineral aggregate and 21.9 percent voids in the mix. The average percentage of voids in the mineral aggregate

<sup>9</sup> Researches on Bituminous Paving Mixtures, PUBLIC ROADS, vol. 7, no. 10, December 1926.

TABLE 10.—Characteristics and stability of cylinders of series 1

Identification	Original material			Distillation residue		Asphaltic residue	
	Stability at 77° F.	Float at 77° F.	Furol viscosity at 122° F.	Stability at 77° F.	Float at 122° F.	Stability at 77° F.	Penetration at 77° F.
A.....	175	9	94	1,100	57	4,125	87
B.....	275	20	205	1,625	110	4,775	81
C.....	100	2	68	775	59	3,400	95
D.....	200	9	199	750	60	3,100	88
E.....	200	36	294	400	31	3,650	104
F.....	200	20	94	375	34	5,250	101
G.....	150	8	68	875	62	5,875	82
H.....	300	65	297	1,000	75	5,525	92
I.....	325	75	320	800	57	5,075	99
K.....	475	100	478	825	73	5,250	100
L.....	275	24	274	750	51	4,375	93
M.....	200	7	151	675	66	4,150	112
N.....	350	48	677	800	62	3,750	105
O.....	225	25	300	375	30	4,025	101
P.....	175	12	201	900	82	4,000	107
R.....	375	17	735	750	50	4,375	102
S.....	150	35	178	525	46	3,375	106
T.....	225	46	300	475	50	3,500	102
V.....	225	52	458	775	59	3,575	99
X.....	125	8	111	1,050	101	3,950	104
Y.....	175	21	281	1,300	122	4,000	85
Z.....	300	53	613	1,575	123	4,400	86

was 33.5 percent for the cylinders containing distillation residues and 32.9 percent for those containing asphaltic residues.

The results of the stability tests on cylinders in series 1 are given in table 10 and of those on series 2 are given in table 11. All results are the average of three cylinders.

**STABILITY OR BONDING STRENGTH DEPENDS NOT ONLY ON THE CONSISTENCY BUT ALSO ON THE INHERENT PROPERTIES OF THE ASPHALTIC BINDER**

The results given in table 10 are shown graphically in figure 18. The stability at 77° F. of the cylinders made with the original materials is plotted against the Furol viscosity at 122° F. and the float test at 77° F. The stability of the cylinders containing distillation residue is plotted against results of float tests at 122° F. on the distillation residues. In the case of the cylinders containing asphaltic residues, since the consistencies of the bitumen are all approximately the same, the stability values are plotted for each sample independently.

Examination of these figures shows that the stabilities developed by the sand mixes in which the original material and distillation residues were used as binders are roughly proportional to the consistency of the contained bitumen and that, for the same viscosity, (Furol at 122° F.) the high-gravity materials give higher stability than the low-gravity materials. In general the asphaltic residues of the high-gravity materials yield the higher stabilities. In the graph showing the stability of the original material plotted against the time of float at 77° F. it is seen that for the same time of float the high-gravity materials do not give the higher stabilities. Since these high-gravity materials have higher float-test values at 77° F. for a given viscosity at 122° F. than the low-gravity materials, the time of float determined at the temperature at which the stability test is made may be a better index of the bonding strength than the Furol viscosity test run at a higher temperature.

Examination of the figure shows that the stabilities and consequently the workability of mixtures made with samples I and R are about the same but there is a difference of over 400 seconds in their Furol viscosities at 122° F. Samples K and V have about the same viscosity at 122° F. but, as measured by the float test at 77° F., sample K is twice as viscous as sample V. The mixture made with sample K has much the greater stability and it would be more difficult to process on the road.

In order to eliminate materials which are difficult to process at normal temperatures, some States have found it necessary to include in their specifications a maximum float-test value at 77° F. on the original material as well as a Furol viscosity at 122° or 140° F. There is no doubt that materials of the same consistency as measured by the viscosity at 122° F. and by the penetration at 77° F. of the asphaltic residues, have different binding values and that the seven high-gravity materials included in this study have the greater bonding strength for equivalent consistencies as measured by viscosity or penetration of the asphaltic residue.

While the variations in stabilities produced by the original materials may be due largely to variations in

TABLE 11.—Characteristics and stability of cylinders of series 2

Identification	Stability at 77° F. of original material	Oven exposed cylinders						Atmospheric exposed cylinders						Distillation residue cylinders		Asphaltic residue cylinders	
		Stability at 77° F. after exposure for—			Percentage loss of bitumen in—			Stability at 77° F. after—			Percentage loss of bitumen in—			Stability at 77° F.	Theoretical loss of bitumen	Stability at 77° F.	Theoretical loss of bitumen
		1 day	7 days	14 days	1 day	7 days	14 days	5 weeks	10 weeks	15 weeks	5 weeks	10 weeks	15 weeks				
A.....	175	300	350	475	6	12	16	700	1,150	1,225	13	18	20	1,125	21	3,525	35
B.....	275	475	750	1,000	6	13	17	1,100	1,575	1,550	15	18	21	1,475	21	4,050	28
C.....	100	175	400	550	11	23	27	600	925	1,300	25	27	28	675	26	2,950	34
D.....	200	325	375	650	7	12	16	800	1,125	1,650	18	19	21	750	18	2,925	25
E.....	200	200	225	325	2	3	6	325	700	1,050	6	9	11	400	9	3,075	34
F.....	200	225	200	425	2	4	8	650	1,475	2,775	9	13	17	375	12	4,575	36
G.....	150	175	275	475	3	8	13	675	1,875	3,400	14	19	23	850	23	4,975	36
H.....	300	425	575	975	1	4	7	1,125	2,775	4,675	8	12	16	925	12	4,650	25
I.....	325	450	675	1,125	2	4	7	1,375	3,175	4,050	8	10	14	800	10	4,900	24
K.....	475	625	825	1,250	3	3	5	1,375	3,325	4,250	5	9	12	875	9	5,625	22
L.....	275	325	525	575	4	8	10	675	1,150	1,500	10	13	16	750	16	3,775	35
M.....	200	275	500	700	6	11	14	825	1,475	2,075	15	18	20	700	20	3,650	35
N.....	350	425	700	1,000	4	7	10	950	1,575	2,125	8	10	11	750	11	3,525	30
O.....	225	250	400	500	3	5	10	700	1,025	1,400	8	11	12	375	12	3,475	35
P.....	175	300	700	925	6	12	17	1,000	1,950	2,550	17	20	21	925	21	3,550	32
R.....	375	450	650	975	4	5	9	875	1,225	2,150	6	8	10	625	9	3,825	29
S.....	150	175	325	525	4	8	13	700	1,375	1,850	11	15	16	475	16	2,950	35
T.....	225	275	450	625	4	7	11	775	1,525	1,975	9	12	13	500	14	3,325	31
V.....	225	300	475	775	3	6	9	875	1,675	2,050	6	10	11	700	12	3,550	28
X.....	125	225	425	675	7	14	20	850	1,875	2,300	19	24	26	1,025	28	3,700	34
Y.....	175	300	500	750	6	9	15	1,100	2,250	2,700	15	19	20	1,225	23	3,800	29
Z.....	300	475	775	1,000	5	8	11	1,350	2,575	2,700	10	14	15	1,425	17	4,300	24

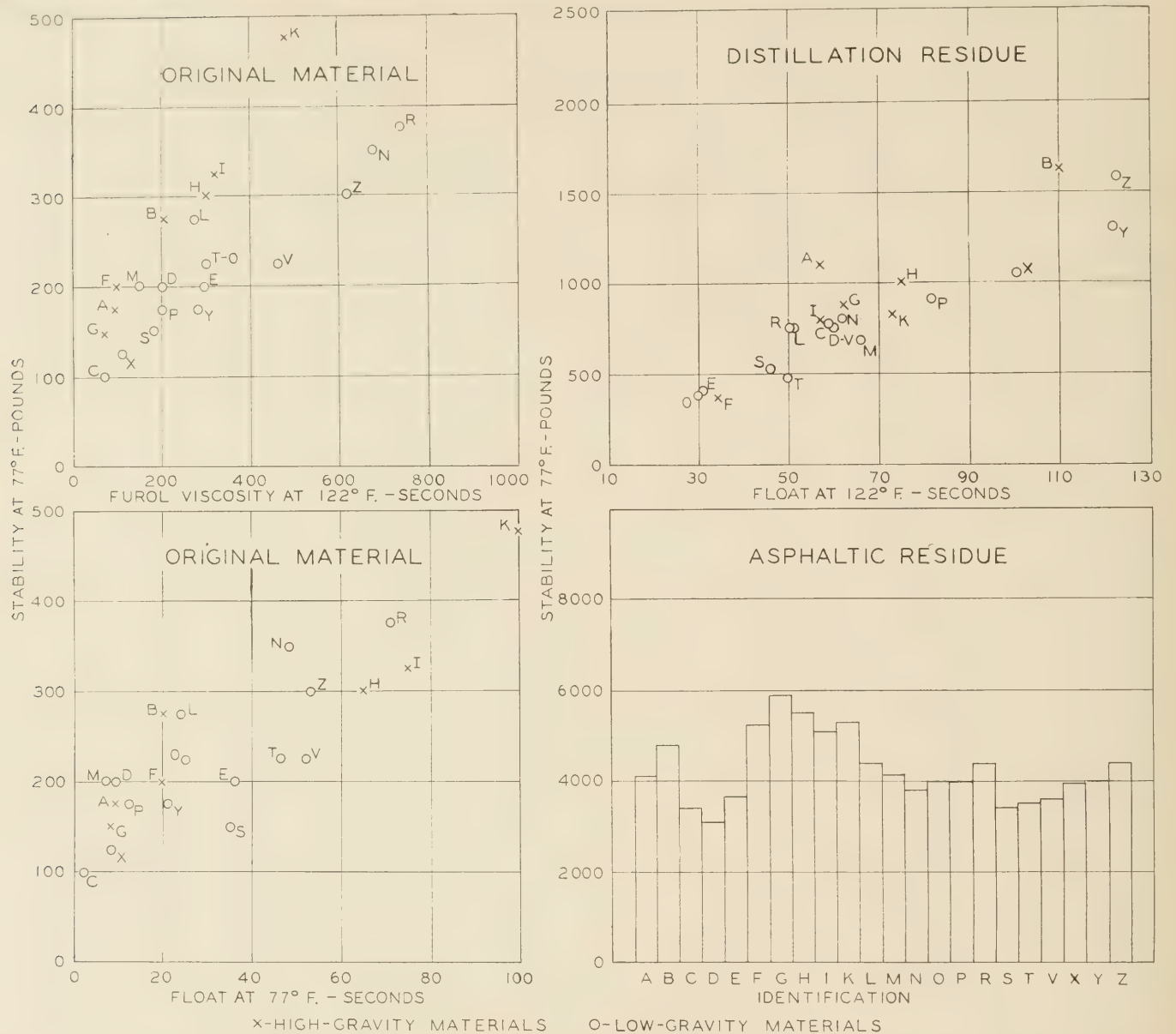


FIGURE 18.—RELATION BETWEEN THE CONSISTENCIES OF ORIGINAL MATERIALS, DISTILLATION RESIDUES, AND ASPHALTIC RESIDUES AND THE STABILITY AT 77° F. OF CYLINDERS OF SERIES 1.

consistencies, the wide differences in stability produced by asphaltic residues having relatively small differences in penetration seem to indicate that factors other than consistency influence the bonding strength of these semisolid residues.

In figure 19 the loss of bitumen in the cylinders exposed in cases for 5 weeks is plotted against the loss of bitumen in the cylinders cured in an oven for 2 weeks. The stability of the exposed cylinders is plotted against the stability of the oven cylinders. Although the loss of bitumen is about the same for both methods the stability of the cylinders exposed for 5 weeks is, in all cases except 2 (samples N and R), greater than the stability of the oven-cured cylinders. Just as changes not entirely due to loss of volatile matter produced hardening and inherent changes in the exposed films, so here greater bonding strength or stability is developed in the exposed cylinders although the loss of volatile matter is practically the same under both conditions of treatment.

In figure 20 the data for the cylinders of series 2 containing distillation residues and cylinders exposed for 5 weeks are plotted in the same manner. Although in all cases the distillation-residue cylinders represent a greater percentage of loss of bitumen than the actual loss occurring in 5 weeks of exposure, the stabilities of the exposed cylinders are greater than the stabilities of the distillation-residue cylinders except in 9 cases and, of those 9 cases, 7 of the materials lost at least 6 percent more in the distillation test than did the cylinders exposed for 5 weeks.

Similar comparisons may be made for cylinders exposed for 15 weeks and cylinders containing distillation and asphaltic residues. However, comparisons are made in a different manner in figure 21 where the stability and the percentage of loss of bitumen of the exposed cylinders and the theoretical loss of the cylinders containing residues are plotted for each sample. It is seen that the stabilities of cylinders exposed for 15 weeks are much greater than the stabilities of the cylin-



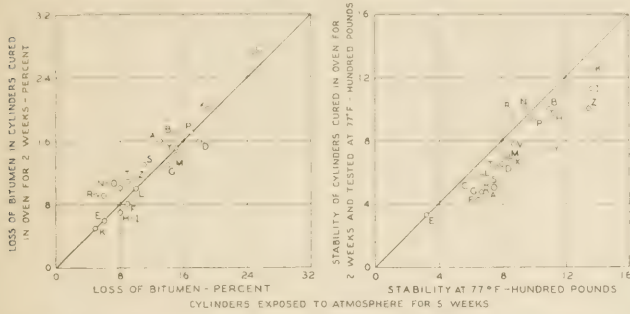


FIGURE 19.—COMPARISON OF LOSSES AND STABILITY OF CYLINDERS SUBJECTED TO OVEN CURING FOR 2 WEEKS WITH THOSE OF CYLINDERS EXPOSED TO ATMOSPHERE FOR 5 WEEKS.

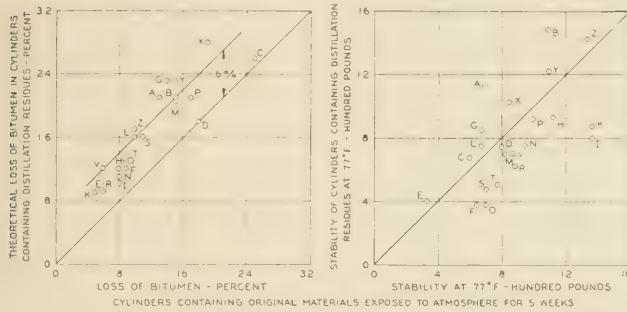


FIGURE 20.—COMPARISON OF LOSSES AND STABILITY OF CYLINDERS EXPOSED TO ATMOSPHERE FOR 5 WEEKS WITH THOSE OF CYLINDERS CONTAINING DISTILLATION RESIDUES OF SERIES 2.

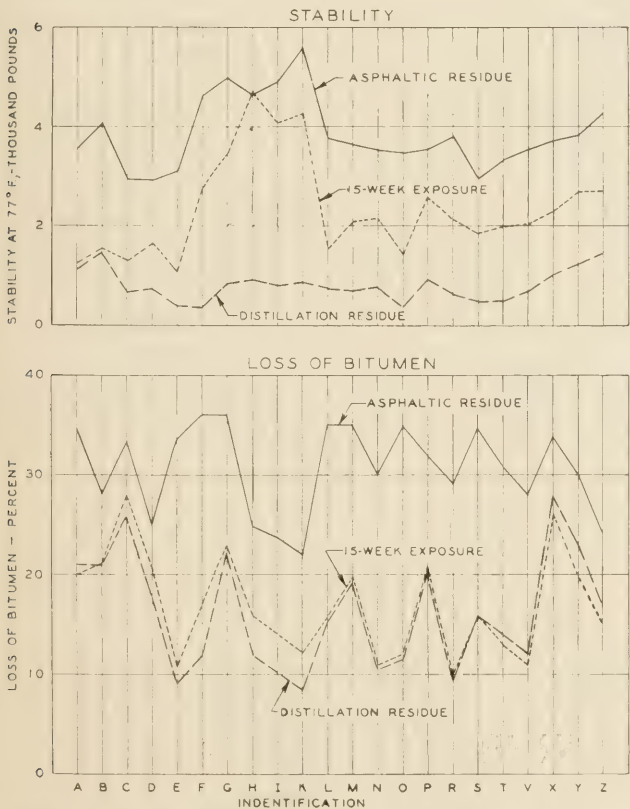


FIGURE 21.—COMPARISON OF LOSS OF BITUMEN AND STABILITY OF CYLINDERS EXPOSED TO ATMOSPHERE FOR 15 WEEKS AND CYLINDERS CONTAINING DISTILLATION AND ASPHALTIC RESIDUES OF SERIES 2.

ders containing distillation residues except in the case of samples A and B. These are 2 of the 3 samples which were altered to the greatest extent in the thin film exposures. The loss in the samples exposed for 15 weeks is about equal to the loss in the distillation test, being a little higher for samples F, H, I, and K but not enough to account for the much higher stabilities obtained in the cylinders of these materials exposed for 15 weeks. The stabilities of the cylinders exposed for 15 weeks do not reach the stabilities of the corresponding cylinders containing asphaltic residues except in the case of sample H, which was one of the materials having the highest gravity, and neither does the loss of bitumen in the cylinders exposed for 15 weeks approach the loss in the asphaltic residue test.

It has been held by some that a certain amount of material insoluble in naphtha is essential if an asphaltic material has or is to develop bonding strength. It is of interest to note that the high-gravity materials which have the greatest amount of material insoluble in naphtha, originally have higher stabilities than the low-gravity materials of the same viscosity at 122° F. and, with the exception of samples A and B, develop the highest stabilities after 15 weeks exposure.

In connection with these comparisons of stability, it should be remembered that the cylinders made with residues contained material of uniform consistency distributed evenly throughout the entire mass. When the cylinders were exposed to atmospheric and oven volatilization the evaporation of the volatile matter undoubtedly was greatest at the surface of the specimens and the cylinders after various intervals of exposure no doubt had a bituminous binder of variable consistency. Differences in consistency of bituminous material throughout the mass of the exposed cylinders may be considerable during the early stages of the exposure. Figure 22 shows the percentage of loss in thin films of bitumen compared with the loss in cyl-

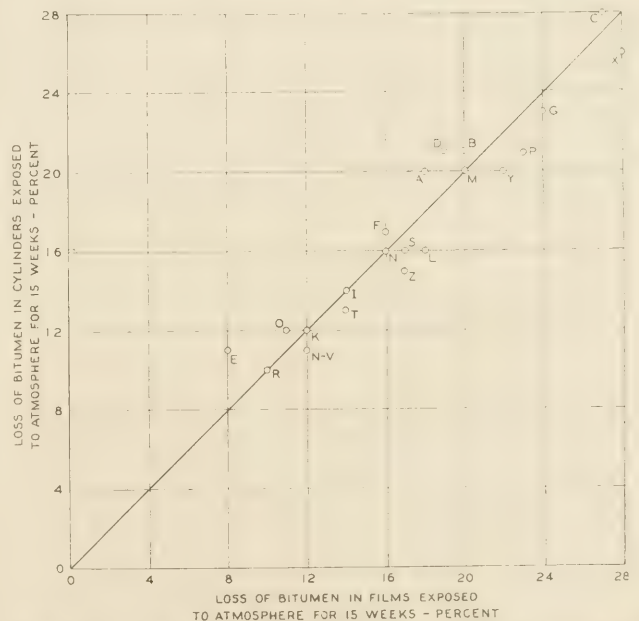


FIGURE 22.—LOSS IN THIN FILMS OF BITUMEN EXPOSED FOR 15 WEEKS COMPARED WITH LOSS OF BITUMEN IN CYLINDERS EXPOSED FOR SAME TIME.

inders exposed for 15 weeks. There is a remarkable agreement between the volatility of the various bituminous materials under widely different conditions. It is therefore concluded that the development of increased stability or bonding strength in the exposed cylinders is caused by the same conditions which are responsible for the greater hardening and changes in the inherent structure of the bituminous materials when exposed in the thin films to atmospheric conditions.

#### SUMMARY OF CONCLUSIONS

It is realized that the exposure of thin films of the original materials and the exposure of the materials admixed with a mineral aggregate in the form of cylinders are not comparable to service conditions; but bituminous surfaces containing materials similar to these indicate the beginning of deterioration by incipient surface failures. The effects of weathering on the original materials and mixtures of original materials with mineral aggregate should prove of value in anticipating service behavior. The tendency of road surfaces to ravel and dust, the necessity for the use of additional oil in reprocessing, and the frequency of retreatments or reprocessing required are all related to the weather resisting properties of the bituminous binder.

This investigation was undertaken to determine the adequacy of present laboratory tests in determining the weathering characteristics of slow-curing liquid asphaltic road materials. The work is being continued with materials from other sources and processes and, while at this time no particular laboratory tests or combinations of tests are recommended, the following conclusions based on the data developed seem justified:

1. The high-gravity materials and their residues which were studied are more susceptible to temperature changes than the low-gravity materials and their residues with the exception of the materials A and B.

2. For materials from the same source the amount of asphaltic residue generally increases with increase in Furol viscosity and generally decreases with increases in distillation and volatilization loss. Certain exceptions to this relation have been noted.

3. The asphaltic-residue test, when used alone, does not indicate the probable rate of loss of volatile matter

in the material under exposure even though the time of reduction to the specified penetration is considered.

4. The distillation test more nearly indicates the ultimate loss which occurs in thin films and mixed samples exposed to the sun and air. The relative rates of volatilization of the various materials investigated can more readily be anticipated from the distillation curves than from the oven volatilization test or the asphaltic residue test alone.

5. There is more artificial hardening due to causes other than loss of volatile matter, in samples exposed to atmospheric conditions than in samples submitted to the various laboratory heat tests.

6. The development of a ductile residue in the asphaltic residue test does not indicate that the material will develop a ductile residue when exposed to the sun.

7. Changes in inherent characteristics, which may be attributed to chemical action, namely, oxidation, polymerization and carbonization, take place to the greatest extent under exposure to the sun and least during distillation.

8. Carbonization does not occur during laboratory heat tests or outdoor exposure, as made in this study, in those materials which originally have little or no matter insoluble in carbon tetrachloride. It does occur in materials which initially have some material insoluble in carbon disulphide and appreciable amounts insoluble in carbon tetrachloride. Specifications calling for high solubility in carbon disulphide and carbon tetrachloride will be advantageous in minimizing the possibilities of carbonization playing a part in the weathering of the materials in road service.

9. The bonding strengths of the original materials are roughly proportional to their consistencies.

10. Asphaltic residues of approximately the same penetration, when admixed with a standard sand, do not always have the same bonding strength or stability.

11. None of the laboratory tests now used to indicate probable service behavior produce residues which either alone or in mixtures are as much altered as are those developed under the exposure as made in this investigation.

12. The bonding strength or stability of mineral aggregate mixed with liquid asphaltic materials of the kind studied depends upon characteristics which cannot be ascertained under the present method of testing.



CURRENT STATUS OF U.S. PUBLIC WORKS ROAD CONSTRUCTION AS PROVIDED IN TITLE II, SECTION 204 OF THE NATIONAL INDUSTRIAL RECOVERY ACT

CLASS II—PROJECTS ON EXTENSIONS OF THE FEDERAL-AID HIGHWAY SYSTEM INTO AND THROUGH MUNICIPALITIES

AS OF MAY 31, 1934

Table with columns: STATE, PUBLIC WORKS FUNDS ASSIGNED FOR CLASS II PROJECTS IN MUNICIPALITIES, COMPLETED (Total cost, Public works funds, Regular Federal aid, Mileage, Estimated total cost), UNDER CONSTRUCTION (Public works funds, Regular Federal aid, Percentage completed, Mileage), APPROVED FOR CONSTRUCTION (Public works funds, Mileage), and BALANCE OF PUBLIC WORKS FUNDS AVAILABLE FOR NEW CLASS II PROJECTS.

TOTALS row: 114,962,369; 10,636,191.26; 116,678.53; 320.0; 73,130,094.50; 70,616,182.12; 363,444.62; 1,106.3; 18,477,694.82; 382.9; 15,447,677.00

**CURRENT STATUS OF U.S. PUBLIC WORKS ROAD CONSTRUCTION  
AS PROVIDED IN TITLE II, SECTION 204 OF THE NATIONAL INDUSTRIAL RECOVERY ACT**

**CLASS III—PROJECTS ON SECONDARY OR FEEDER ROADS**

AS OF MAY 31, 1934

STATE	PUBLIC WORKS ROAD CONSTRUCTION FOR CLASS III PROJECTS ON HIGHWAYS		COMPLETED		UNDER CONSTRUCTION			APPROVED FOR CONSTRUCTION		BALANCE OF FUNDS AVAILABLE FOR NEW CLASS III PROJECTS		STATE
	Total cost	Mileage	Total cost	Mileage	Estimated total cost	Public works funds allotted	Percentage completed	Mileage	Public works funds allotted	Mileage		
Alabama	\$ 2,092,533.00	0.1	\$ 10,175.87	0.1	\$ 366,618.51	\$ 366,618.51	19.9	26.8	\$ 1,162,648.19	91.4	\$ 553,090.43	Alabama
Arizona	629,435.00	6.0	40,700.00	6.0	427,644.64	415,517.65	42.5	28.9	66,882.80	7.4	102,334.55	Arizona
Arkansas	1,687,084.00	32.0	32,039.78	9.6	849,463.55	849,463.55	24.9	102.2	185,100.21	27.0	620,460.46	Arkansas
California	3,369,691.00	355.0	355,186.02	7.9	2,699,373.23	2,459,826.28	34.4	146.0	569,125.32	22.8	295,908.80	California
Colorado	1,716,532.00	436.0	436,510.43	74.3	1,135,694.96	1,100,233.89	55.4	100.2	13,641.00	1.2	108,949.96	Colorado
Connecticut	659,120.00				684,915.58	659,120.00	17.3	14.5				Connecticut
Delaware	454,772.00				181,790.00	181,790.00	28.6	1.8	72,847.50	12.2	200,434.50	Delaware
Florida	1,307,958.00				962,433.87	962,433.87	64.2	49.3	28,739.81	2.5	46,199.68	Florida
Georgia	2,320,573.00				892,163.25	892,163.25	37.4	72.4	319,217.64	30.1	1,039,948.88	Georgia
Idaho	1,424,566.00				841,478.63	786,135.47	51.7	98.8	610,178.46	21.1	49,591.98	Idaho
Illinois	5,265,960.00				4,296,284.50	4,296,284.50	26.8	26.3	28,739.81	5.7	46,199.68	Illinois
Indiana	501,892.00				462,227.52	462,227.52	26.6	63.4	39,664.48	5.7	4,950.00	Indiana
Iowa	2,212,295.00				1,506,756.47	1,434,700.00	45.8	167.4	661,295.00	95.3	4,950.00	Iowa
Kansas	2,522,401.00				2,236,457.37	2,225,500.00	36.2	156.2	1,331,879.45	12.2	10,304.84	Kansas
Kentucky	1,879,340.00				1,551,784.50	1,530,399.25	52.1	177.2				Kentucky
Louisiana	1,457,148.00				755,309.16	755,309.16	17.5	35.1	359,846.18	8.9	226,365.01	Louisiana
Maine	842,479.00				415,452.89	329,202.47	90.0	35.3	311,197.00	20.2	7,948.21	Maine
Maryland	891,132.00				522,207.30	504,281.30	52.4	40.5				Maryland
Massachusetts	488,185.00				469,741.41	469,741.41	54.3	15.2	28,000.00	1.0	18,443.59	Massachusetts
Michigan	3,184,057.00				2,788,300.00	2,788,300.00	22.4	216.3	64,197.67	4.0	238,257.00	Michigan
Minnesota	2,376,415.00				1,760,625.95	1,742,450.25	17.0	213.5	29,000.00	1.0	228,573.93	Minnesota
Mississippi	1,744,869.00				692,710.94	692,710.94	40.3	76.0	215,115.72	28.4	836,242.34	Mississippi
Missouri	2,923,275.00				2,607,472.61	2,607,472.61	71.0	462.9	191,946.91	10.2	5,611.96	Missouri
Montana	1,859,337.00				1,242,345.11	1,242,345.11	6.5	142.4	41,036.97	4.1	26,572.33	Montana
Nebraska	1,957,040.00				1,927,606.96	1,870,877.77	52.4	229.4	20,499.00	4.0	2,700.49	Nebraska
Nevada	1,136,479.00				821,374.65	821,374.65	60.7	88.1				Nevada
New Hampshire	477,460.00				518,207.67	477,460.00	69.0	25.6				New Hampshire
New Jersey	56,950.52				56,950.52	56,950.52	94.4	.5	44,141.06	1.9	349,093.41	New Jersey
New Mexico	1,448,254.00				779,017.95	779,017.95	61.9	236.9				New Mexico
New York	3,608,768.00				3,787,900.00	3,350,250.00	23.4	88.6				New York
North Carolina	2,380,571.00				801,953.56	800,776.92	42.1	88.2	406,891.66	49.3	588,439.94	North Carolina
North Dakota	1,451,112.00				3,269,130.00	2,994,354.90	37.8	169.8	278,150.00	92.3	924,952.07	North Dakota
Ohio	3,871,148.00				3,269,130.00	3,269,130.00	37.8	169.8	278,150.00	92.3	124,765.10	Ohio
Oklahoma	2,304,199.00				1,402,135.25	1,394,165.61	19.8	122.5	992,276.44	148.6	17,756.95	Oklahoma
Oregon	1,566,724.00				1,366,366.73	1,330,342.61	31.3	77.8	191,946.91	15.3	29,128.08	Oregon
Pennsylvania	7,344,822.00				6,769,344.52	6,653,681.12	35.8	609.7	460,239.17	44.7	160,805.71	Pennsylvania
Rhode Island	499,677.00				412,465.08	412,465.08	38.3	33.2	180,080.44	20.6	87,211.98	Rhode Island
South Carolina	1,364,791.00				1,155,839.36	1,155,839.36	52.3	133.1	259,134.52	96.7	9,724.48	South Carolina
South Dakota	1,502,470.00				330,244.31	330,244.31	41.5	114.5				South Dakota
Tennessee	2,123,159.00				1,003,653.16	1,003,653.16	22.3	80.8	594,059.12	46.9	441,145.03	Tennessee
Texas	6,061,006.00				1,404,019.32	1,404,019.32	41.2	470.0	151,045.64	6.5	44,128.10	Texas
Utah	1,048,677.00				433,810.37	414,465.50	25.4	52.2	102,614.44	8.2	41,925.32	Utah
Vermont	438,860.00				407,794.94	407,794.94	25.3	33.1	51,378.13	3.3	45,853.85	Vermont
Virginia	1,699,900.00				1,204,794.96	1,204,794.96	52.3	231.5	224,277.35	1.3	9,724.48	Virginia
Washington	1,080,673.00				894,022.41	894,022.41	33.8	45.4	271,739.30	3.9	6,483.69	Washington
West Virginia	1,118,559.00				909,293.97	909,293.97	29.8	50.7	132,079.37	9.1	71,205.70	West Virginia
Wisconsin	2,431,220.00				1,953,899.48	1,843,520.84	25.7	140.6	207,404.19	22.2	149,350.60	Wisconsin
Wyoming	1,125,332.00				650,533.25	650,533.25	35.0	92.7	103,202.00	16.4		Wyoming
District of Columbia	959,234.00				848,409.36	848,409.36	38.9	5.7			205.30	District of Columbia
Hawaii	1,871,106.00				1,171,117.69	1,171,117.69	23.2	4.9			9,368.31	Hawaii
<b>TOTALS</b>	<b>92,931,302.52</b>		<b>9,164,565.95</b>	<b>1,442.9</b>	<b>67,324,881.03</b>	<b>64,572,070.94</b>	<b>37.5</b>	<b>5,831.0</b>	<b>9,968,944.12</b>	<b>1,010.9</b>	<b>9,225,721.51</b>	<b>TOTALS</b>





