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A vast amount of research work has been done in an endeavor to improve this situation and numerous test methods and procedures have been tried in search of an answer to the problem of establishing quality tests for asphalt.

Perhaps at this point it will be helpful to trace briefly something of the history of established test procedures. To a large degree these are either a rationalization of preconceived ideas with mechanically improved means for standardizing rule-of-thumb methods, or are developments of analytical procedures typical of general laboratory practice.

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DEPARTMENT OF PUBLIC WORKS
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EARL WARREN, Governor
C. H. PURCELL, Director of Public Works

QUALITY TESTS FOR ASPHALTS

A Progress Report

By F. N. HVEEM
aff Materials and Research Engineer

DECEMBER, 1946

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Quality Tests for Asphalts—A Progress Report

By F. N. HVEEM, Staff Materials and Research Engineer

ANYONE having more than a casual knowledge of the behavior and characteristics of asphaltic paving materials is aware that most of the standard tests do not indicate the actual quality or suitability of asphalts for road building purposes. Asphalts which meet all of the usual specification requirements may prove to be more or less unsatisfactory and, conversely, cases have been known where materials which do not meet orthodox test requirements have given excellent performance.

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Perhaps at this point it will be helpful to trace briefly something of the history of established test procedures. To a large degree these are either a rationalization of preconceived ideas with mechanically improved means for standardizing rule-of-thumb methods, or are developments of analytical procedures typical of general laboratory practice.

In the days when the design of asphalt paving mixtures rested largely in the hands of construction superintendents or "hot stuff" foremen, i. e., the type of individuals usually designated as "practical men" it required considerable experience and the exercise of some ingenuity in order to detect differences in character of materials. Asphalts were, undoubtedly, subject to at least as many variables 40 years ago as at present, and a paving man would often shape a small piece of paving asphalt and stretch it by hand in order to determine if the material was "short." From observing this practice Dow developed the ductility machine making it possible to stretch a sample of asphalt under controlled conditions. (At the present time, however, it appears to be very difficult to establish any simple relationship between the results of such ductility tests and performance.)

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This edition has been revised and corrected in the light of more recent experience.

TEST METHODS

Many "old timers" could make a very good estimate of consistency by chewing a sample of asphalt and then someone employed a needle to test the softness. (It is reported that in former days a No. 2 sewing machine needle was specified.) This expedient was, of course, a forerunner of the present penetrometer.

A second group of test methods has grown out of standard laboratory practices, such as fractionation by distillation and by determining the so-called solubility in certain solvents.

The majority of engineers have rather taken it for granted that all of these tests rest upon well-founded and proven bases. It would be illogical and unfair to be critical of the pioneers who devised our present standard laboratory procedures; they deserve every credit, but progress usually involves casualties in time-honored methods and viewpoints and we need not assume that the existing standards are sacred. Familiar things are usually taken at their face value without too much questioning or probing beneath the surface to understand the true significance or meaning. This tendency may account for the lack of realistic quality tests for asphalts. The real properties of materials and causes of certain phenomena are not always immediately evident and the investigator can take nothing for granted in trying to find "what causes a good asphalt pavement."

SOME QUESTIONS

Before trying to develop quality tests it might be well to decide just what qualities *are* important. In other words, what must be expected of a paving asphalt? What is its function and what properties are essential and which unimportant?

To the chemist, asphalt is a mixture of hydrocarbons mutually dissolved and dispersed. To the highway engineer, asphalt is something used to stick particles of sand, gravel or crushed stone together in order to form a pavement which will withstand the abrasion of automobile tires, resist deformation under the weight of the vehicle, and withstand the deteriorating effects of rain and sun.

It was once thought that the stability of a pavement depended on the hardness of the asphalt, but as it has been widely demonstrated that stable surfaces can be constructed with practically any grade ranging from 20 penetration to an SC-2 road oil, and as all grades of asphalt have been found adequate to resist ravelling or abrasive action (provided proper proportions are used), it appears that *any adhesive semiliquid material which can be made sufficiently fluid to permit mixing with the aggregate, which will adhere well to the stone in the presence of water, and which will not become brittle with age, will make a suitable binder for a road surface;* and it does not matter whether it be asphalt, rubber, tar, or molasses, if it has these properties and is cheap enough.

Thus we can conclude that only four primary properties need be determined by testing, all others are irrelevant or comparatively unimportant. These properties are consistency, durability, rate of curing, and resistance to water action.

Consistency

The engineer must have some means for classifying bituminous materials of varying fluidity or hardness and for this purpose standard test methods are reasonably adequate. Undoubtedly, they can and will be improved. It would be desirable if all asphaltic materials could be compared on the same numerical scale and it would be nice if we could deal in fundamental units and express viscosity in poises or centipoises. Nevertheless, this is a refinement and the problem of determining consistency is not clamoring for immediate solution.

It should be recognized, however, that consistency measurements may serve two purposes. First, the viscosity of the product must be taken into account in considering the type of construction operation. Road mixing, for example, is only feasible with the more liquid grades and is ordinarily impracticable with anything heavier than Grade 4 products of either slow, medium, or rapid curing types. In the plant mixing process consistency can be altered at will through the application of heat.

MATTER OF CONSISTENCY

Secondly, choice of a certain consistency depends upon conditions after construction. The character of the aggregate gradation may influence the designer in selecting consistency. For example, a Grade 2 liquid asphalt would not ordinarily be suitable for macadam aggregate and hard paving asphalts have many drawbacks when mixed with very fine materials. (The surface area of the aggregate should be taken into account when selecting the most appropriate grade of asphaltic binder.) A third consideration involves the permanence of the construction. For example, it is often desired that the bituminous surfacing be susceptible to reworking or reshaping at intervals and obviously only the more liquid asphalts should be used in such cases.

It may then be concluded that the consistency of asphaltic products is important under two quite distinct conditions and, therefore, under two different temperature ranges. It appears that serious attention should be given to the desirability of reporting consistency of all asphaltic materials at a temperature appropriate for mixing and construction operations and at a temperature typical of service conditions. It is recognized, of course, that actual working temperatures vary considerably. Nevertheless, it would be enlightening, for example, to indicate not only the consistency at a temperature between 70° F. and 100° F. but also at another point between 200° F. and 300° F.

Admitting that it is possible to distinguish between the various degrees of fluidity by using available testing equipment the next problem to be considered is whether or not the asphaltic material will retain its original properties and it is the ability of an asphalt to remain unchanged which determines the degree of durability.

Durability

Before discussing the durability of asphalts, it must be emphasized that all failures of bituminous pavements can not be charged to the asphaltic binder. There is a definite tendency for many engineers to blame any troubles on the asphalt or else to some vaguely inferred "chemical action." But before seeking to cure the ills of bituminous pavements by adjusting the properties of the asphalt, we must identify those troubles *for which the asphalt alone is responsible.*

A careful study of the types of distress which can be shown to occur with one asphalt and not with another of the same grade indicates that the most frequent and consistent difference to be observed is in cracking of the pavement, and/or dusting and disintegrating of the surface from abrasion. Failures and deterioration of this type can be pretty broadly covered by recognizing that the asphaltic binder has become too hard or too brittle and has lost the properties of plasticity, ductility or malleability. Test procedures aimed at measuring durability have been of two general types. One group we may designate as typical of the chemist's approach while the other group represent physical tests which may require the use of mechanical devices.

LABORATORY OPERATIONS

Laboratory operations which involve the analytical procedures typical of the chemist's profession are the various solubility tests. For example, the solubility in CCl_4 and CS_2 undoubtedly have the general purpose of detecting asphalts which had been damaged by overheating in the process of manufacture and perhaps these tests are useful for the purpose.

In California experience, however, after testing many thousands of samples we have no record of a sample of commercial asphalt which failed to pass, but, nevertheless, we have received unsatisfactory asphalts. Furthermore, any sample which contains a percentage of mineral matter as in the case of most natural bitumens would not meet the standard requirements for solubility but a small amount of inert mineral matter can hardly be considered detrimental as filler dust is deliberately added as a component of most dense paving mixtures. These tests cannot be considered, then, as reliable indications of quality. (It is also obvious that the engineer should not be too greatly concerned with refining practices provided that the ultimate product is suitable for the purpose intended.)

SOLUBILITY RELATIONSHIPS

The solubility in petroleum ether and the various modifications of the Oliensis Spot Test are also attempts to classify asphalts by their so-called "solubility relationships." Whether or not this group of tests indicates the presence of asphaltenes or "heterogeneity" or whether they simply indicate something of the equilibrium of a colloidal system in the presence of organic fractions depends largely on the viewpoint and the background of the individual.

The second group of tests deals with the vapor pressure relationships and includes the flash test, the loss on heating, distillation, and the retention of consistency after a sample has been heated. All of these methods or analytical procedures have shown some correlation with performance and undoubtedly there are a number of measurable differences between a group of "good" asphalts and "poor" asphalts.

There is, however, one fatal weakness in virtually all these tests which is undoubtedly the principal reason for the lack of correlation which is known to exist. This is due to the incontrovertible fact that all of the test methods are performed on samples which are maintained in a fairly large bulk with a very limited area of exposed surface and for the most part evaporation and hardening tests are conducted at elevated temperatures. In contrast, asphalts in the pavement exist largely as films representing a vast surface area compared to the volume of asphalt and rarely are heated above 140° F. There is ample evidence to prove that hardening and changes in asphalt are largely a surface phenomena and marked differences have been reported due to variations in temperature and agitation in the presence or absence of oxygen.

There is no question that analytical methods have their value. Undoubtedly, they will continue to be used but the consumers and users of asphalts are desperately in need of means which will detect durable asphalts from nondurable, irrespective of the reasons for the difference. As nondurable asphalts fail through becoming brittle and chalky and by losing their ability to resist pounding and abrasion it seems an obvious conclusion that durability tests should establish the existence or absence of these properties after the asphalt has been subjected to conditions typical of its service life. Such a durability test will be described later in this paper.

Rate of Curing

A third property of asphalt which is of concern to the consumer is the rate of curing of the more liquid products. These road oils and cutbacks have been logically classified as slow curing, medium curing, or rapid curing, and there are times and places where one or the other of these is most appropriate and definitely superior to the other alternates. Nevertheless, there is a considerable variation at times in products purchased under one of these designations. Materials have been delivered which met all of the test requirements for a slow-curing product and yet which were found to set up so rapidly that road mixing became difficut if not impossible.

On the other hand, rapid curing cutbacks have been specified and purchased under standard specifications but which were found to set up or cure no more rapidly than the medium-curing grades. It is clearly evident that existing specifications which undertake to control rate of curing by limitation of the distillation test are not adequate for the purpose and it seems reasonable to assume that failure to correlate is due to the marked difference between test conditions and temperature and those of service.

Again, in order to simulate service conditions, the asphalt must be spread in thin films and subjected to moderate temperatures and as curing obviously involves increasing viscosity or hardening of the asphalt, all that is necessary is to measure this change under appropriate conditions. A procedure for accomplishing this will be described later under the section devoted to recommended or proposed tests.

Resistance to Water Action

A fourth property of asphalts which will be of some concern to the user is the ability to adhere well to mineral aggregates and to resist stripping or separation by the action of water. It is not believed that asphalts ordinarily differ widely in this respect. It is generally known that the greatest variable exists in the mineral aggregates. Nevertheless, the user would like to know that an asphalt has at least the average capacity to stick in the presence of water.

The foregoing completes the summary and discussion of the essential properties of asphaltic materials so far as the user is concerned. Beyond a doubt the producer of asphalts may consider many other factors and conditions in manufacturing an asphalt which will meet quality tests. If tests can be established which deal only with essential properties, the purchaser and user of asphalts need not concern themselves with source, method of manufacture, or composition of the substance. In other words, if the material has the properties to make a good durable pavement, the consumer should not care who makes it or how or from what ingredients.

Research dealing with the above problems has been under way for some time in the California laboratory and is by no means completed and no implication is made that it is entirely original. If we have made any progress, we must acknowledge a considerable debt to all other effective workers in the field. The following will describe such progress and conclusions as have been made in answer to the four problems just outlined.

Consistency

Only a limited amount of work has been done on new methods of measuring viscosity or consistency. As previously mentioned, it is felt that this is the least acute and it is possible to get along quite well by the use of existing methods. It is believed that this phase of testing ultimately will be clarified by the adoption of true viscosity measurements applied to all grades ranging from 40 penetration to grade "O" liquid asphalt.

Interesting results have been secured by use of the Brookfield Synchro-lectric Viscosimeter. This apparatus is particularly attractive because of the possibility of measuring viscosity at a number of temperatures in a comparatively short time by a simple apparatus. However, the equipment should be modified and could doubtless be improved for routine work.

We are aware of the work by other laboratories using a modified Ostwald type of Viscosimeter**(1)** but the method appears too complex and time-consuming to be the ideal answer for a large consumer laboratory.

Durability Tests

The matter of durability has seemed to be the most pressing. A considerable amount of work has been carried out. Satisfying the conditions set forth in the previous discussion of the durability problem we have made numerous attempts to measure the behavior of asphalt in thin films. Two methods suggest themselves.

First, to spread the asphalt in a thin film over a plane surface, such as a glass or metal. Benson**(5)** developed a technique for spreading small smears on microscope slides and the success reported by Benson in classifying the films by microscopic examination after exposure to radiant energy tends to confirm the soundness of the belief that thin films are essential. We followed Benson's lead for some time and reached the conclusion that while informative and vastly interesting it, nevertheless, does not lend itself well to specification purposes. The patterns developed represented a too wide variety for easy classification.

Adopting the mechanical approach, similar smears were made on strips of thin sheet metal which after exposure to heat and light were subjected to abrupt bending and observed for cracking.

SECOND ALTERNATE

A second alternate was the use of thin films on circular tinned metal discs, such as lids of three-ounce ointment tins. After weathering, the film was subjected to sudden impact through the dropping of a steel ball on the reverse side of the tin. This produced a raised, rounded bump under the asphalt causing cracks in a brittle film and by making a series of these tests after a number of weathering cycles, it was possible to determine the effect of weathering in producing hardening. The primary difficulty was in establishing films of uniformly even thicknesses and it was quickly demonstrated that the resistance to shatter or cracking varied markedly depending on the thickness of the film. We were unable to devise any means for spreading these films with absolute accuracy and uniformity over the entire surface. The method has definite possibilities if this difficulty can be overcome.

Another approach is to utilize a standard aggregate and this thought immediately brings to mind Ottawa sand. At this point in our investigation, the work reported by Lang and Thomas **(3)** came to hand and we immediately tried out certain of the methods described in this report. Three test procedures on Ottawa sand-asphalt specimens were reported by Lang. One, an elongation test, requires the manufacture of a special briquette which is pulled apart in a stretching device. The prospect of manufacturing these specimens as a routine operation was definitely unattractive; therefore, this operation was rejected.

LANG METHOD

A second method used by Lang with which we also experimented involves the manufacture of a briquette of Ottawa sand and asphalt which is destroyed by the impact of a hammer. This procedure has two definite weaknesses. One, the difficulty of detecting precisely when the first evidence of fracture has occurred. Second, the impossibility of gauging the weight of the blow. This means that a number of specimens must be tested involving a considerable amount of work, time and material.

The abrasion method however, has none of these limitations and as Lang's report seemed to indicate that the correlation by the abrasion method was at least as good as by any other, work was undertaken using the Deval machine as indicated by Lang. This was soon dropped in favor of a special tumbling device designed for the particular purpose. Many trials were made with generally gratifying results. It was clearly evident that very high losses were developed in specimens known to contain poor asphalts while relatively small losses were found with known good materials. Attempts were made to standardize and improve the reproductibility of results. However, while the test results were generally in the right order, it was found impossible to reproduce values on identical specimens as closely as could be desired.

Studying the problem further, the thought occurred that specimens could be subjected to abrasion by a stream of sand falling freely from a prescribed height. Simple trials were made and the method immediately indicated marked differences between briquettes manufactured from cracked asphalts as compared to good grades of steam-refined products. The difficulty in uniformly compacting a briquette one inch in depth and two inches in diameter lead to the adoption of a relatively thin specimen. The study has resolved itself to the method now in use.

This consists of preparing a mixture of Ottawa sand with 2 percent of the asphalt under test using a mixing temperature of 230° F. For the lower penetration grades, the lowest possible mixing temperature (above 230° F.) is used. (Mixing temperatures can, of course, be established as desired. For example, it may be most logical to use temperatures typical of paving plant practice for the particular asphalt.) Sufficient mix is prepared to make a number of 30 gram specimens which are compacted immediately in the form of small plaques using the lids of three-ounce ointment tins for the purpose. **(Figures 1A, 4 and 5)** These lids will hold approximately 30 grams of mixture when compacted level full. The samples are spread out in a flat pan and cured in a special weathering machine which employs an atmospheric temperature of 140° F. during which samples are exposed to the direct rays of standard drying lamps which emit the bulk of their energy in the infrared band. A circulation of air is maintained. One complete cycle in this machine requires about five hours. The essential elements of this weathering operation could be produced very cheaply in a simple device built along the lines of the Weatherometer using radiant energy in the infra-red and a circulation of air.

TESTING APPARATUS

Two samples are tested immediately and others are removed from the curing ovens at intervals of three cycles and tested for abrasion loss under the impact of a stream of 10-14 mesh steel shot. The testing apparatus is so constructed that the specimen is rotated under a stream of shot falling freely through a tube 2½ cm. in diameter and 1 meter in length **(Figures 2 and 3)**. These dimensions are arbitrarily selected and are not necessarily the most ideal. Steel shot is allowed to fall at the rate of 1,800 grams per minute and the wear on the test specimen is expressed in grams lost per 1,000 grams of abrasive. A 10-14 mesh steel shot has been used because of its ready availability and ease with which it may be reclaimed after being mixed with the asphalt coated particles which are eroded from the specimen.

ABRASION TESTS NOT NEW

The use of this technique has proved to be the most satisfactory thus far considered and has the advantage of utilizing comparatively small quantities of materials for the test, specimens are easily prepared, large numbers can be handled in a weathering device of reasonable size, the tests are quickly made. Reproductibility is excellent and marked differentiation is shown between samples of known good and bad performance. Questions remaining to be finally settled are the appropriate temperatures for most significant results and the type and the development of a variable temperature laboratory mixer which will approximate the mixing conditions found in the field.

Abrasion tests or sand blast tests are not new. We have reports of a sand blast method used in Germany a number of years back and Rhodes of the Koppers Company **(6)** has reported favorably on the use of a sand blast device for measuring the durability of tar-sand specimens.

In adopting our present procedure it was felt that utilizing a gravitational fall would be much more simple and more easily controlled than the use of an air blast with the sand. In all cases we are strongly influenced by the consideration that to become a satisfactory routine method a test procedure should be as simple as possible, should preferably involve no expensive apparatus and be capable of being made in large numbers.

DEGREE OF WEATHERING

The principal drawback to this method is that a period of several days is involved as tests must be made after six or nine cycles of weathering in order to establish clear differences. The most satisfactory and significant degree of weathering is yet to be definitely fixed upon.

Figure 6 indicates the range of values developed by a series of asphalts ranging from good to poor based on service performance.

Figure 7 is a chart representing the durability rating of a series of asphalts which represent straight run steam refined, cracked, also cracked and blown.

Figure 1 represents three plaques or test specimens of compacted Ottawa sand-asphalt mixtures weighing approximately 30 grams, compressed under 1,000 lbs. per square inch using the lid of a standard three-ounce gill ointment tin to hold the specimen.

Specimen A represents the specimen appearance as initially compacted showing no loss under sand abrasion. Specimen B is a specimen showing moderate loss. Specimen C indicates a considerable loss typical of an asphalt lacking in durability.

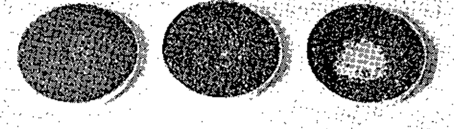
Figure 2 is a schematic diagram of the durability test apparatus for subjecting specimens (illustrated in Figure 1) to the abrasive action of sand falling through a tube one meter in length. Test specimen is rotated and entire system subject to temperature control.

Figure 3 is a photograph of the apparatus diagrammed in Figure 2.

Figure 4 shows the units used in preparing the durability test specimens.

Specimen A is the plunger or follower as commonly used in the Hubbard-Field Test, dia. 1.994 inches. Specimen B is a cylindrical steel mold 2 inches inside diameter. Specimen C is a lid of a standard three-ounce ointment tin, diameter 2-3/16 inches. Specimen D is a base plate with grooved recess to hold the tin lid. Specimen E is the funnel for introducing Ottawa sand-asphalt mixture.

Figure 5 is the same as Figure 4, showing apparatus assembled, ready for applying compression load of 3,500 pounds total (i. e. 1,000 lbs. per square inch).



A B C
Figure 1

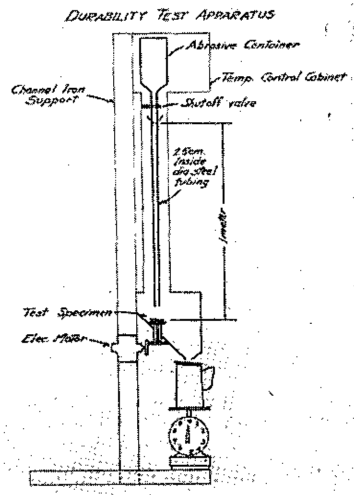


Figure 2

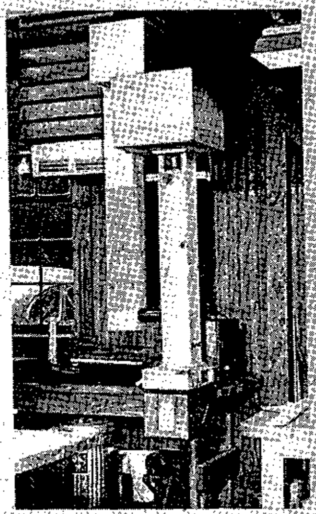
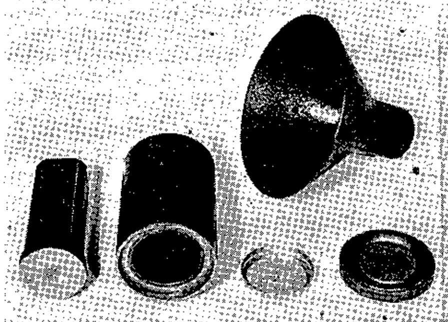


Figure 3



A B C D E
Figure 4

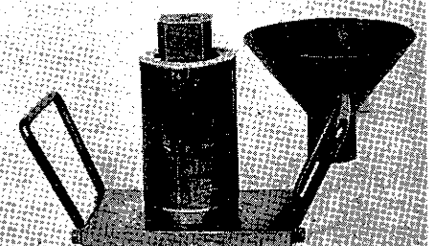


Figure 5

Table I gives detail of other test data. While no service record is available it is believed, however, that the data are significant as the durability test shows all steam refined asphalts to have low loss and, hence, good durability, while cracked asphalts show an increased susceptibility to abrasion loss and cracking plus blowing is apparently even more detrimental. It will also be noted that the Mid Continent crudes may not be adversely affected by a certain degree of cracking if not carried too far.

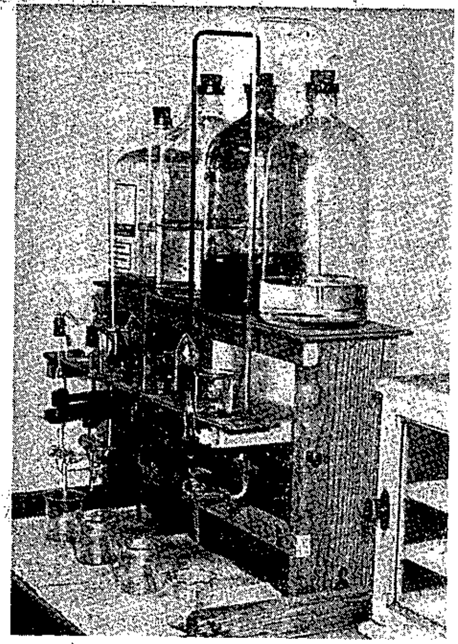
MECHANISM OF HARDENING

Figure 9 represents durability results on seven samples of SC oils from the State of Wisconsin all of which met Standard Specification requirements according to Mr. Zapata. He advises that No. 1 and No. 7 have given poor service. **Figure 9** indicates the poor quality of No. 7 while **Figure 21** seems to account for No. 1.

A test of this type does not carry any particular implication or produce direct evidence as to the mechanism of hardening. It has been variously maintained that the hardening of asphalts is due primarily to the loss of volatiles **(2)**. Others contend that oxidation is a primary factor. Polymerization and thixotropy have been mentioned and no one can question that all of these mechanisms may play a part. Oxidation tests such as the Kansas City Testing Laboratory Oxidation **(11)**, as well as those reported by Nicholson **(7)** and Skidmore **(8)**, Raaschig and Doyle **(9)**, Anderson, Stross and Ellings **(10)**, all have shown a considerable degree of correlation with performance.

Even though hardening may develop through several types of mechanism, there seems to be good reason for finding out something about the capacity of an asphalt to take up oxygen and it appears unquestioned that if an asphalt will combine with any appreciable quantity of oxygen, its physical characteristics will be changed.

Working in the California Laboratory, Mr. A. R. Ebberts devised a technique for quantitative measurement of the relative oxidation of asphalt in thin films **(4)**. Details of this method have been previously reported but



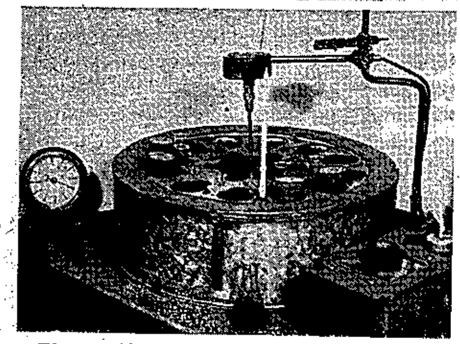


Figure 10. An automatic pipette layout for dispensing re-agents. Figure 11. The water bath maintained at 140 degrees Fahrenheit for the conditioning of specimens.

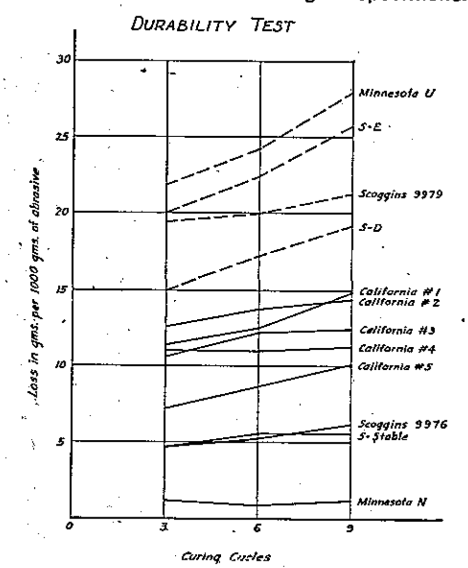


Figure 6 is a chart showing abrasion loss on 12 samples of asphaltic materials which have given previous indications of quality either in service or by extensive laboratory investigations. Samples N and U, furnished by the Minnesota Department of Highways, were previously reported by Lang and Thomas.[3] Samples marked S-stable, S-D and S-E furnished by Mr. V. A. Endersby of the Shell Development Company, were classified as follows:

S-stable, carefully refined asphalt found to be durable on field experiments; Samples S-D and S-E were cracked to varying degrees; and S-E was found to be very unstable and ravelled badly on the street under moderate traffic. Samples Nos. 9976 and 9979 were furnished by B. I. Scoggin of the Anderson-Prichard Oil Corporation. Sample No. 9976 was reported as showing excellent performance in pavements over 11 years old. Sample No. 9979 was a product of a Winkler Koch cracking unit and has not proved to be satisfactory in service.

The samples marked California No. 1, 2, 3, 4, 5 are typical California 85-100 penetration asphalts manufactured from various California crude oil sources.

modifications have been made and at present details of the test procedure are being studied in order to make it possible to obtain satisfactory reproducibility between operators.

There is some evidence that satisfactory asphalts should have neither too great nor too little capacity to combine with oxygen. Furthermore, the oxygen demand evidently varies with

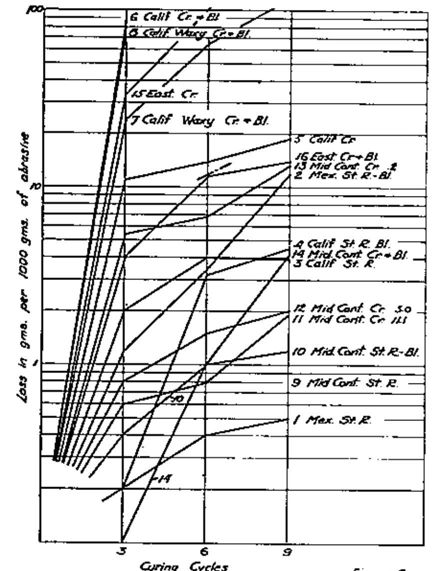


Figure 7

the different grades and a consistent increase may be traced from the heavier paving grades to the most liquid materials. For example, it appears that satisfactory asphalts of 50 penetration should consume not less than 0.9 ml. nor more than 1.2 ml. of $KMnO_4$

TABLE I

A.	B	C	D	E	F	G	H	I	J	K
Sample No.	Source of Crude	Str. Run	Cracked	Blown	A.P.I.	Pen.	Sft. Pt. Ring & Ball	Suscept. Index	Ebberts Oxid.	Abrasion
1	Mexican	x	------	------	------	103	119.5	2	4	1
2	"	x	------	x	------	100	144	1	8	9
3	Calif. Lube	x	------	------	------	97	110	15	5	7
4	"	x	------	x	------	94	112	10	3	8
5	"	------	x	------	------	108	108	16	9	12
6	"	------	x	x	------	107	110	9	15-16	15
7	Calif. Waxy	------	x	x	------	92	112.5	8	14	13
8	"	------	x	x	------	100	113	5	15-16	16
9	Mid-Cont.	x	------	------	------	103	112	7	1	2
10	"	x	------	x	------	84	118	3	2	3
11	"	------	x	------	11.1	108	109.5	12	6	4
12	"	------	x	------	5.3	92	112	13	7	5
13	"	------	x	------	—.2	96	111	12-13	10	
14	"	------	x	x	5.3	92	116	4	10	6
15	Eastern	------	x	------	------	105	110	11	12-13	14
16	"	------	x	x	------	86	115.5	6	11	11

The above table lists certain data on 16 samples of asphalts representing different crude sources and different methods of treatment in the refinery. Column B indicates source of crude, Columns C, D, and E indicate the refinery process, and columns F, G, H, and I indicate A.P.I. Gravity, Penetration, Softening Point, and Susceptibility Index (according to Piper and Van Doormal). Column J indicates the relative order of quality as indicated by the Ebberts Oxidation Test. Column K indicates the relative order of quality indicated by the Durability test at 6 cycles as shown on Figure 7.

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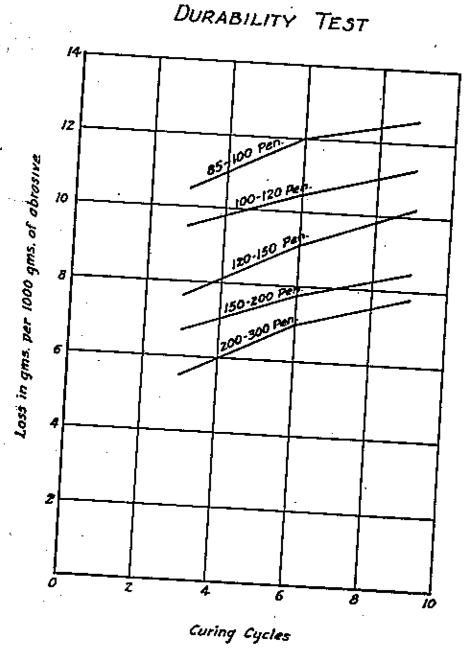

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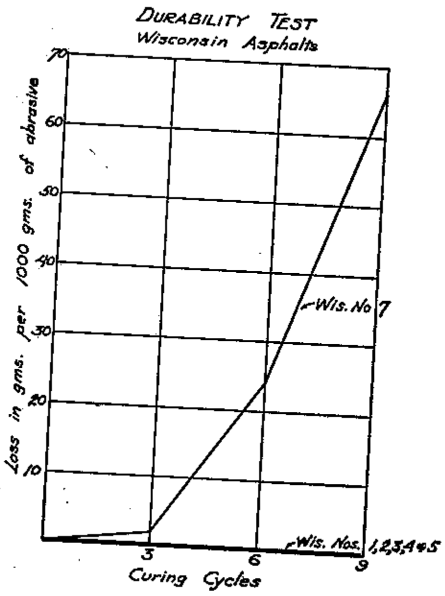

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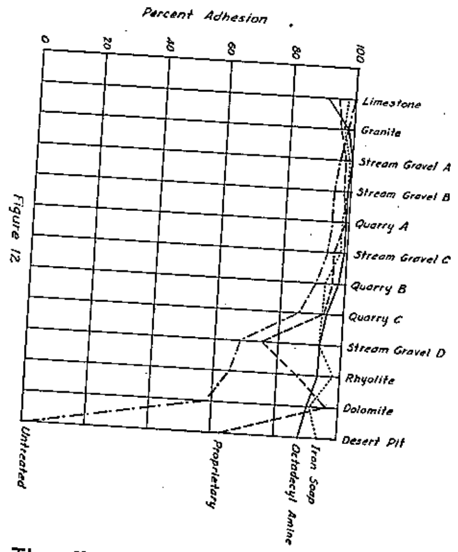

The effect of various wetting agents as assessed by the dye adsorption technique is shown graphically in Figure 12. An adhesion of about eighty-five per cent (85%) is necessary to assure satisfactory service life. These agents seem to be most effective when the aggregate is damp at the time of mixing. The mixtures were all made with SC-3 oil.

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Surprising uniformity has been secured, although occasional increments may run out of line. It should be possible to prevent these occasional discrepancies, however, they are not serious as the large number of breaks makes it possible to virtually eliminate errors by averaging the individual lengths. To all intents and purposes this particular machine produces from six to 20 test results on each specimen in the short space of a few minutes **(Figure 18)**.

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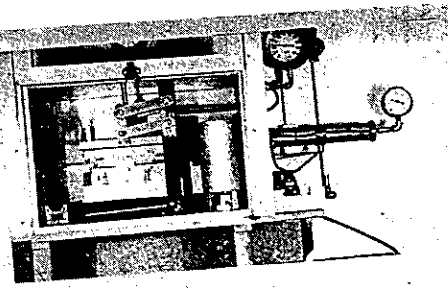


Figure 13

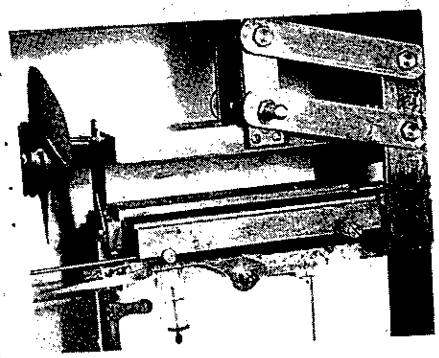
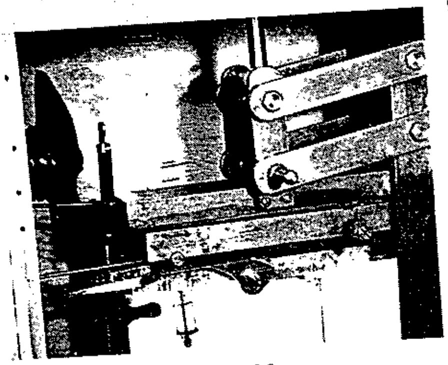


Figure 14



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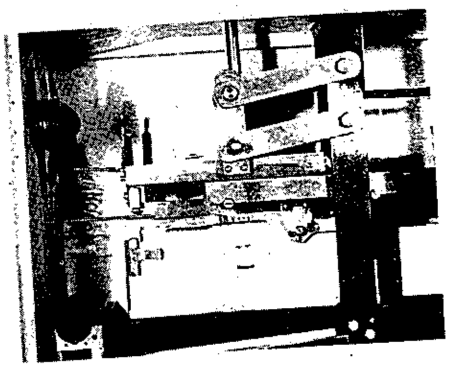
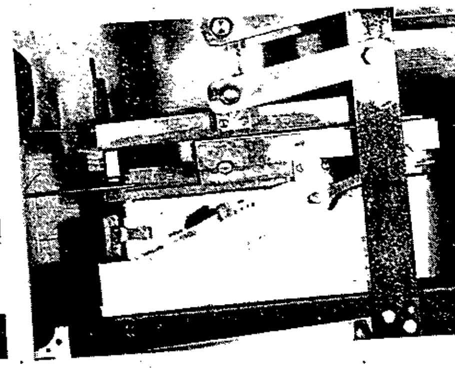


Figure 16



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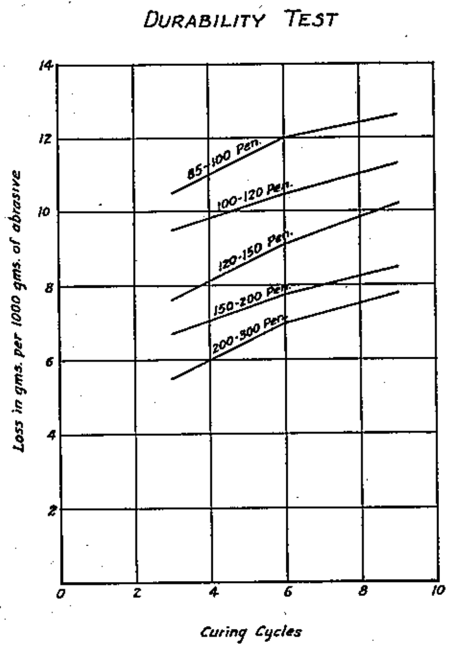

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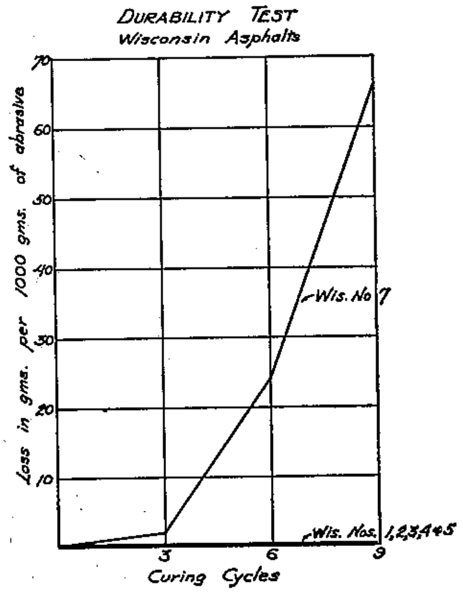

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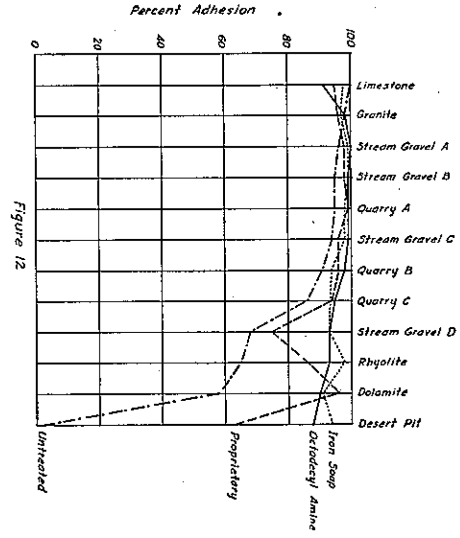

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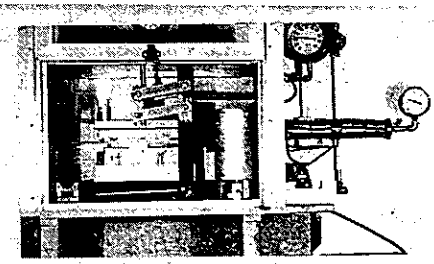

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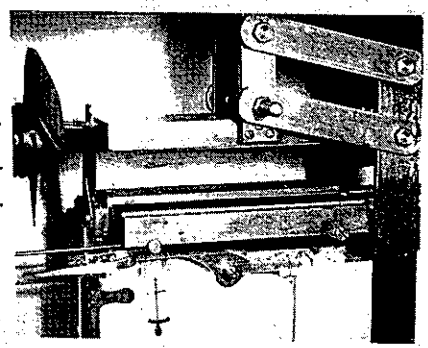

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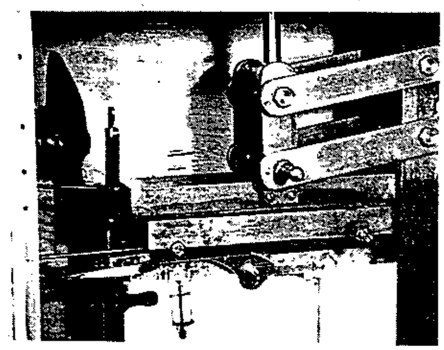

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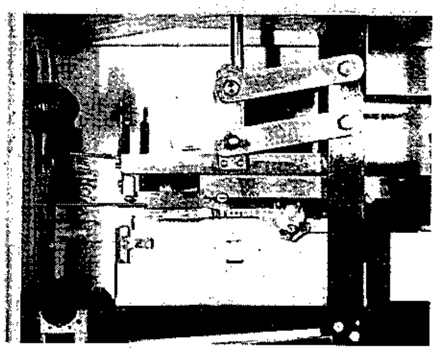

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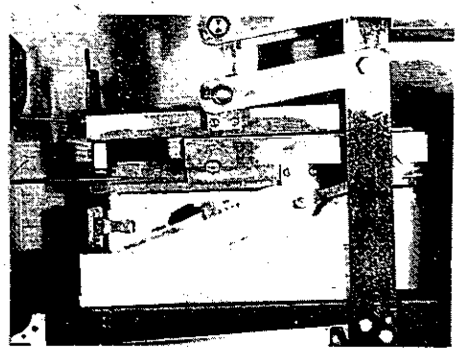

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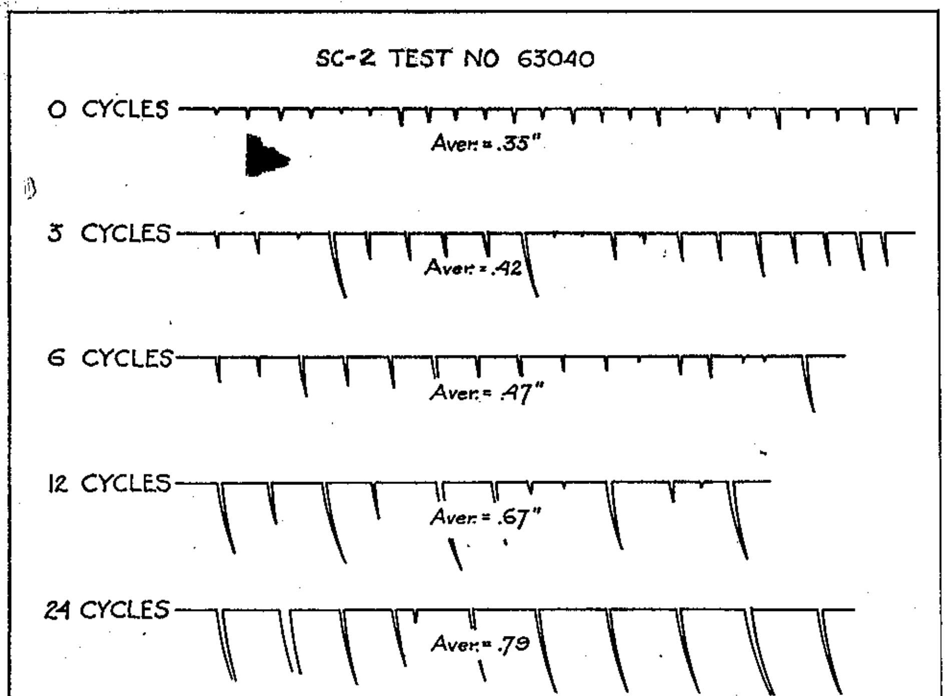

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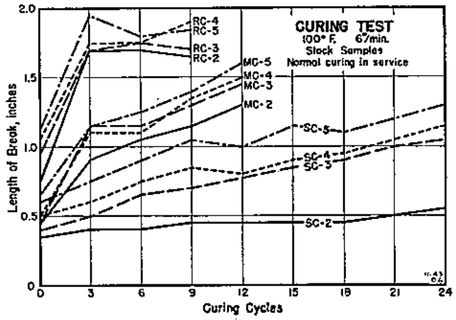

Figure 19 is a chart showing relative behavior of liquid asphalts in the curing test after a varying number of cycles of curing. These products have all given normal performance in service so far as rate of curing and final consistency is concerned. The chart indicates that curing characteristics may be identified after only three cycles of accelerated curing. While minor discrepancies exist, the pattern of behavior for slow curing, medium curing, and rapid curing are distinctive and the several grades fall approximately in normal sequence.

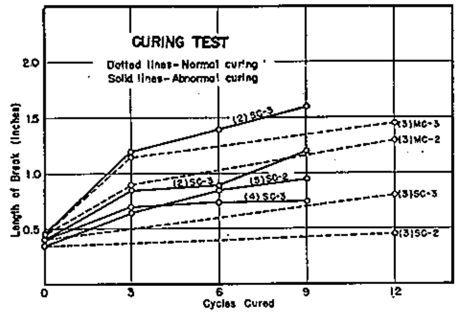

Figure 20. A chart showing several oils wherein the curing characteristics are at marked variance with the normal for the grade. The dotted lines indicate normal curing behavior for the grades indicated. The solid lines show the rate of curing of certain products which have been observed to perform in an erratic manner during construction operations. These test data seem to be in good agreement with observed performance.

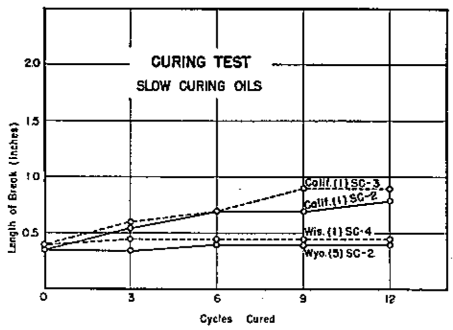

Figure 21. This chart illustrates the difference in test results between slow curing and non-curing samples. Wisconsin No. 1 and Wyoming No. 5 were reported as failing to set up or harden on the road. Two California oils which have shown normal curing are included for comparison.

the desired speed. While a hydraulic cylinder was used in the original machine to permit ready variation of speed, this type of drive will not be satisfactory for routine control after a standard speed has been agreed upon.

Thus we have two methods, both dealing with the asphalt spread in films over the surfaces of aggregate particles and subjected to the hardening influences of radiant energy and oxygen. The methods are simple and direct.

Resistance to Water Action

The fourth property of asphalts concerns the resistance to water action. While tests for this type of failure have been in use since 1929 in the form of swell tests and stripping tests, there is still much to be desired in the way of correlation with actual performance. While these tests so far have generally been used to classify the mineral aggregates there is no doubt that asphalts also vary somewhat in their ability to adhere in the presence of moisture.

Work has been under way in the California laboratory for some time seeking to develop a quantitative measure of stripping. This work is based on the known fact that most surfaces will adsorb dyes of various types and by selecting a suitable dye it was hoped that the amount of stripped surface could be accurately measured. The procedure followed consists of, first, determining the dye adsorption of the untreated aggregate. For this purpose the entire material is used ranging from four-mesh to and including the dust. This constitutes a definite improvement over the standard stripping test as the smaller sizes are generally the most important due to the large surface area represented. The aggregate is then coated with a standard grade of asphalt and after curing over night it is subjected to the stripping test. Dye is then added and the adsorption again determined.

PERCENTAGE OF STRIPPED AREA

By comparing the amount of dye adsorbed after the stripping test has been performed to the amount taken up by the entire aggregate before being coated with asphalt, it is possible to determine the percentage of stripped area. This type of determination has shown good reproducibility but requires the attention and time of an operator who is well-trained in the techniques employed. The method cannot be recommended or advocated in its present state as a routine operation but does provide a means for obtaining comparative data on adhesion phenomena **(Figure 12)**.

If a dependable method can be devised which will show good correlation with field performance, it might then be possible to classify asphalts by using some standard aggregate for the purpose. It is to be feared that adhesion tension relationships between asphalts and aggregates may be specific rather than general and if this proves to be the case, the classification of asphalts by means of a single standard aggregate would not be very useful. At present, it appears necessary to use several aggregates of varying composition in order to get a fairly reliable picture of the ability of asphalt to stick.

A complete solution to this entire problem may readily appear from another source. There are now many methods of treatment being advocated or in process of development which should improve the capacity of any asphalt to stick to virtually all aggregates under the most adverse conditions. When an agent is commercially available which can be added to the asphalt at the refinery, this entire problem may largely disappear, although it is likely that accurate test methods will always be needed in order to compare the effectiveness of competing forms of treatment and to assure ourselves that the wetting agent is actually present in specified quantities in the delivered material.

At the present time, three experimental projects are being constructed at selected points in Districts II, VIII and XI, in order to observe the effectiveness of several additives under actual roadway conditions. However, final conclusions cannot be drawn for some time.

RECOMMENDATIONS

With perfection and further corroboration of the results indicated in the foregoing, the next step would be the inclusion of such quality tests in all Standard Specifications. It is suggested that a typical specification would consist of limitations on the flash point corresponding to present practice, specifications for viscosity or penetration and maximum percentage of water. It would then appear to be in order to discard all requirements dealing with distillation, residue of 100 penetration, tests on the residue from distillation and solubility of CCl_4 and SC_2, as well as the xylene equivalent or spot test. In addition, the tests for paving asphalt, loss on heating for five hours at 325° F. and penetration after loss on heating could also be dropped.

In lieu of these requirements, limits of performance under the curing test would indicate a maximum and minimum rate of change for all grades of liquid asphalt. This would serve in lieu of the distillation procedure. Tests for durability would be included, also the test to indicate oxygen demand. It is believed that with the adoption of tests of this type or modifications thereof that the process of selecting suitable bituminous binders would be much simplified and would be in some respects less restrictive than present specifications, but on the other hand would be far more positive in rejecting definitely unsuitable materials.

Evidence thus far available indicates that when judged by these determinations all cracked products are not necessarily objectionable. It appears that a great deal depends on the degree and extent of the cracking and a moderate amount may even be beneficial. This would seem to be in line with reported experience, particularly from the Middle West.

The point should again be emphasized that properly coordinated tests will permit the acceptance of all materials which are capable of producing satisfactory construction and would eliminate all which are unsuitable for the purpose and that having such test procedures the purchaser need not be concerned with either source of material or method of manufacture. The problem of manufacturing a binder or cementing agent which will meet these requirements is up to the producer.

The work described above was carried out in the Laboratory of the Materials and Research Department under the general direction of Mr. T. E. Stanton, Materials and Research Engineer.

It is also desired to acknowledge the valuable contributions and suggestions made by the following individuals who have worked on various phases of the problem.

A. R. Ebberts, Associate Physical Testing Engineer.

Ernest Zube, Associate Physical Testing Engineer.

George Sherman, Assistant Physical Testing Engineer.

John Skog, Assistant Physical Testing Engineer.

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