Technical Report Documentation Page

1. REPORT No.

2. GOVERNMENT ACCESSION No.

3. RECIPIENT'S CATALOG No.

4. TITLE AND SUBTITLE

Proposed New Tests and Specifications for Paving Grade Asphalts

5. REPORT DATE

February 1963

6. PERFORMING ORGANIZATION

7. AUTHOR(S)

Hveem, F.N.; Zube, Ernest; and John Skog

8. PERFORMING ORGANIZATION REPORT No.

9. PERFORMING ORGANIZATION NAME AND ADDRESS

State of California
Department of Public Works
Division of Highways

10. WORK UNIT No.

11. CONTRACT OR GRANT No.

13. TYPE OF REPORT & PERIOD COVERED

12. SPONSORING AGENCY NAME AND ADDRESS

14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

Presented at the Association of Asphalt Paving Technologists Meeting San Francisco, Calif. February 18- 20, 1963

16. ABSTRACT

Anyone interested in the technology of asphaltic paving mixtures is aware of the importance of the asphaltic binder in providing a durable pavement. It is also well known that the standard routine tests do not indicate, in a satisfactory manner, the required engineering properties, and a vast amount of research has been performed in an endeavor to improve this situation.

In recent years a number of investigators have employed the theory of viscoelasticity to explain the mechanical behavior of asphalts. The results of these studies simply confirmed a well known fact: namely, that the mechanical properties of an asphalt are dependent on the temperature and speed of loading. Recognition of these concepts have allowed a better understanding of required engineering properties for paving grade asphalts.

In 1943 this department presented an initial Progress Report on our studies concerned with asphaltic materials. This current report presents results attained since that date, together with a tentative specification for paving grade asphalts that is based on standard and recently developed new test methods.

A description of equipment and test procedures for mixing, weathering and testing Ottawa sand-asphalt mixtures is presented together with studies involving the correlation of these units with field mixing performance and pavement service life.

17. KEYWORDS

18. No. OF PAGES:

109

19. DRI WEBSITE LINK

http://www.dot.ca.gov/hq/research/researchreports/1961-1963/63-14.pdf

20. FILE NAME

63-14.pdf

STATE OF CALIFORNIA
DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS

PROPOSED NEW TESTS AND SPECIFICATIONS FOR PAVING GRADE ASPHALTS

By

F. N. Hveem
Materials and Research Engineer

Ernest Zube
Supervising Materials and Research Engineer

John Skog
Senior Chemical Testing Engineer

63-14

Presented at the Association of
Asphalt Paving Technologists Meeting
San Francisco, Calif. February 18-20, 1963

February 1963

Proposed New Tests and Specifications for Paving Grade Asphalts

By

F. N. Hveem*
E. Zube**
J. Skog***

SYNOPSIS

Anyone interested in the technology of asphaltic paving mixtures is aware of the importance of the asphaltic binder in providing a durable pavement. It is also well known that the standard routine tests do not indicate, in a satisfactory manner, the required engineering properties, and a vast amount of research has been performed in an endeavor to improve this situation.

In recent years a number of investigators have employed the theory of viscoelasticity to explain the mechanical behavior of asphalts. The results of these studies simply confirmed a well known fact; namely, that the mechanical properties of an asphalt are dependent on the temperature and speed of loading. Recognition of these concepts have allowed a better understanding of required engineering properties for paving grade asphalts.

In 1943 this department presented an initial Progress Report on our studies concerned with asphaltic materials.

---

*Materials & Research Engineer, **Supervising Materials & Research Engineer, ***Senior Chemical Testing Engineer, California Division of Highways, Sacramento, California.

This current report presents results attained since that date, together with a tentative specification for paving grade asphalts that is based on standard and recently developed new test methods.

A description of equipment and test procedures for mixing, weathering and testing Ottawa sand-asphalt mixtures is presented together with studies involving the correlation of these units with field mixing performance and pavement service life.

A new type of thin film test for measuring changes in the asphaltic binder during mixing operations has been developed. Studies appear to indicate that this test closely simulates the changes in physical properties of the asphaltic binder during mixing operations.

The development of the Cohesiograph test for measuring the "setting" property of paving grade asphalts is presented together with field correlation studies. The relation of viscosity at 140°F. and 225°F to the "setting" quality is also discussed.

Changes in the physical properties of the binder during service life have been studied by means of the Shot Abrasion Test and the Shell Microviscometer techniques. Modifications in the Microviscometer technique have become necessary in order to attain a more realistic evaluation of engineering properties of the asphaltic binder. The importance of measuring both viscosity and ductility at the end of a designated weathering period is stressed and a micro-ductility unit for measurements of the ductile properties of thin film weathered material is described. The importance of measuring and controlling "shear susceptibility" during weathering is stressed and the need for specification limits is illustrated by field failures caused apparently by lack of control of this very important factor.

The need for control of syneresis of paving grade asphalts is discussed together with studies on the use of the standard ASTM test for measuring the oil exudation property of asphalts.

A tentative specification based on standard and newly developed tests is presented. This specification includes requirements for flash, original consistency, temperature susceptibility, syneresis, "setting" properties, and permissible changes in physical properties during mixing and service life.

INTRODUCTION

During the past twenty five years there has been an increasing amount of research on asphalts. These studies were particularly intensified after the end of World War II, and both consumer and producer groups have contributed in an effort to the better understanding of this complex material. Extended and involved research has been concerned not only with physical tests but also with chemical studies in an effort to unravel the nature of asphalt. Other studies involving the science of rheology have defined the response of the binder to various conditions of loading, and provided fundamental concepts relative to desired engineering properties.

Although the results of these investigations have provided new and important knowledge of the properties of asphalt, there has been virtually no change in specification test methods or requirements for paving grade asphalts during the past twenty-five years. All paving engineers and asphalt technologists agree that the presently used specification tests provide little or no information or real assurance to the engineer who is responsible for the service performance of an asphalt concrete pavement. However, during the recent period of intensive research the only test adopted by the ASTM and AASHO organizations is the Thin Film Oven Test of the Bureau of Public Roads, and this test is the only new one that now appears in the Western States specification. Even this test is still being used only on a regional basis. In our opinion, basic knowledge on the entire subject of asphalt technology is of continuing importance. However, these studies and those performed in the future must start to provide information to the engineer engaged in writing specifications for consumer organizations, as to the permanence and durability of the asphalt.

For many years the Materials and Research Department of the California Division of Highways has been associated with both field and laboratory studies on asphaltic materials. These studies have been mainly concerned with the engineering properties of the binder and little effort has been devoted to separation and study of various chemical groups that constitute the cementing agent. We believe that this phase of the subject is best explored by producers and institutes that have the technical knowledge and trained personnel for these extremely complicated studies. On the other hand the highway engineer is directly concerned with asphalt as an adhesive or cementing agent and his interest in the material is primarily concerned with its ability to properly perform its functions during the service life of the pavement.

In the 1943 Proceedings of this organization, Hveem (1) presented a paper entitled, "Quality Tests For Asphalts - A Progress Report." In the introduction to this paper, the author outlined the required properties of any asphaltic binder as follows: "It appears that any adhesive semi-liquid 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." "Thus we can conclude that only four properties need be determined by testing, all others are irrelevant or comparatively unimportant." "These properties are:

1. Consistency
2. Durability
3. Rate of Curing or Setting
4. Resistance to Water Action."

In paving grade asphalts, Rate of Curing is involved with the "setting" properties of the paving mixture during and immediately following construction. At the present time it is our belief that these four fundamental properties, adequately measured by suitable tests and defined by specifications, will provide a satisfactory adhesive for pavements. This is illustrated in Fig. 1 wherein the part played by the asphaltic binder in the service performance of the pavement is analyzed in terms of the four mentioned properties.

It is readily admitted that the development of adequate tests and the writing of suitable specifications for these properties is a very involved task that will require further field and laboratory investigations. Further, studies presented in this paper clearly indicate the great importance of attaining a proper balance of all engineering properties. The production of an asphalt having exceptional excellence in one characteristic of these properties may lead to a detrimental change in another quality and cause pavement failure or distress.

Our studies to date on the development of tests for measuring these esteemed properties together with necessary specification requirements are described in this paper. The paper should be considered as a continuing progress report of studies subsequent to the 1943 publication.

TESTS

Consistency

Asphalt being a thermoplastic material will vary in consistency with change in temperature. Interest in measuring this property has intensified in the last few years and test methods are now available for determining consistency in absolute units in the range of 40°F to 325°F. There still appears to be a need for better tools to be used in the low temperature range at least in terms of apparatus for routine control work. Another aspect of this problem that requires further study is the expression of the viscosity-temperature relationship. R. L. Griffin, and associates (2) presented an excellent outline of available methods for expressing viscosity over a wide range of temperature. The authors noted the inadequacy of present methods and it appears highly desirable that studies be continued on this important aspect of measuring and evaluating consistency.

"Setting" Property

Certain types of paving mixtures may be difficult to compact properly, and after rolling may remain "tender" for periods of up to two weeks. The inability to roll the mixture at proper temperatures, leads to problems in attaining sufficient density and may require continued rolling for periods of up to one week after completion of the pavement. The "tenderness" of the pavement, immediately after construction produces problems when traffic must be carried through the work. Pavements in such condition tend to "scuff" under turning movements and this may become quite serious on city street intersections, parking areas and service stations. Furthermore, serious problems have recently been encountered in newly constructed parking lots where women's spike heels tend to sink into the pavement.

The California Division of Highways has been concerned with the setting problem since 1940. At that time, we noted that paving mixtures produced in one of our Districts with asphalt from a certain source might be "slow setting" when paving was performed during periods of high day and night atmospheric temperatures. This finding was confirmed in conversations with experienced paving contractors using this asphalt source for parking lots and service stations. In either case, there did not appear to be a problem of mix design or construction procedure, since changing of the asphalt source definitely reduced or stopped the complaints. On the other hand, the same asphalt that caused complaints during high ambient temperatures, did not cause any trouble when atmospheric temperatures were below approximately 80°F.

The problems with "slow setting" paving mixtures in California increased somewhat following the adoption of more restrictive asphalt specifications by the California Division of Highways in 1955. Since this date extensive field investigations have been performed by the Materials and Research Department and the results of this continuing study indicate that there are four basic factors which influence the "setting" characteristics of a paving mixture:

1. Atmospheric temperatures during and immediately following construction.
2. Source of the paving asphalt.
3. Mix design.
4. Traffic during construction. (Need to carry immediate vehicle traffic or pedestrian movement over the new pavement.)

We have noted that these factors are consistently interrelated and involved in the actual field manifestation. As an example, it has been noted by this department and by the

Corps of Engineers, Sacramento District (3) that a "sensitive" asphalt displays "setting" problems in the same paving mixture, depending on the atmospheric temperature during construction. On the other hand, a normal asphalt would not produce a slow "setting" mixture under the same circumstances. These observations have been further substantiated by the recent studies of Woodward (4).

The influence of the asphalt source on the "setting" quality of a paving mixture continues to be a highly debatable subject. R. J. Schmidt and associates in a series of excellent papers (5, 6 & 7) maintain that the role of the asphalt is secondary in comparison with factors involved in the mix design. However, the authors have not explained the fact that all slow "setting" mixtures having a "critical" design will ultimately "set" up even if left completely undisturbed following final rolling. This final "set" will be attained within a week or two after construction and there is most certainly no substantial change in the viscosity of the binder during this short period. The fact that a slow "setting" pavement left in an undisturbed state will "set" up and become resistant to normal service even though inadequately compacted during construction and of a "critical" design, clearly indicates that binder properties are very important in the development of the "setting" property.

Although there are other factors influencing the "setting" properties of a paving mixture, it appears essential to control this property of a paving asphalt in order to eliminate this factor and to insure uniformity of binder behavior during construction. Therefore, intensive laboratory studies were initiated by this department in 1956, with the objective of developing a suitable test for controlling the "setting" property of paving grade asphalts. Laboratory studies were also followed by field correlation work in order to judge the significance of test developments.

In our early field observations of the "setting" property, we noted that asphalts producing a slow "setting" paving mixture were very highly peptized sols displaying almost pure Newtonian behavior even after mixing operations. The asphaltene content was very low leading to a very well solvated system. As expected these asphalts were also quite temperature susceptible. On the other hand, asphalts from other sources that were not associated with troubles even during paving under high atmospheric temperatures were definitely non-Newtonian in their original state and were relatively high in asphaltenes. Rheological data indicate that these asphalts were not too well peptized in the original state and probably in terms of the solution theory would easily separate high molecular weight fractions with the possible formation of "structure". The importance of these findings was increased when field observations indicated that a 200-300 penetration grade from one satisfactory source actually produced better "setting" mixtures than a 60-70 grade from a "sensitive" asphalt source. Although it is readily admitted that consistency at elevated temperatures is an important factor in the "setting" problem the relative ease of formation of "structure" in the asphaltic binder, immediately following placing of the pavement, appears to also be a most significant factor in the differences between "normal" and "sensitive" asphalts.

The above outlined theory was the basis for our development of the Cohesiograph test to measure the "setting" quality of paving grade asphalts. The Cohesiograph apparatus was first described by Hveem (1) for studying the curing rate of liquid asphalts.

An important factor in the development of a test method for measuring the "setting" property is the use of the binder in a physical state closely resembling that found in the paving mixture during placement. These properties, of course, differ from the original material as each asphalt will vary in rate of change in physical properties during mixing. The Ottawa sand asphalt mixes used in the Cohesiograph test are prepared in a laboratory mixer calibrated with field performance, which produces an asphalt at the end of the mixing period that closely resembles the asphalt that is laid in the pavement. The detailed test method is described in Appendix A and briefly outlined as follows: Following preparation of a 2% asphalt-Ottawa sand mixture, the material is compacted into molds. Two specimens are immediately tested in the Cohesiograph at 140°F and the initial or zero hour break length is determined. Two compacted specimens are placed in a 140°F oven and allowed to remain for twenty-four hours. At the conclusion of this period the specimens are again tested in the Cohesiograph and the gain, if any, in the original length of break is determined.

The Cohesiograph test provides two important factors in characterizing the "setting" property of a paving grade asphalt. The first is the original reading which appears to be related to the consistency of the asphalt at the test temperature, Fig. 2. The second factor is the gain in length of break (tensile resistance) after the 24-hour hour heat treatment. This gain, in our opinion, is related to the tendency of an asphalt to develop "structure". Some technologists may argue that this gain is simply caused by hardening of the asphalt or flow of the binder to the contact points of the spherical Ottawa sand grains. However, the remolding of the specimens sharply reduces the 24-hour reading and in some cases reduces it to the original state, Fig. 3. Further, asphalts having poor "setting" qualities are generally quite temperature susceptible and, therefore, any flow of the binder should be expedited. Actually, such asphalts show the lowest gain after the 24-hour heat treatment.

A rather extensive field correlation program has been performed to determine the ability of the Cohesiograph test to detect "sensitive" asphalts in terms of the observed "setting" properties of the paving mixture. The results of this investigation are shown in Figs. 4 & 5. The correlation appears quite satisfactory when considering that only one of the four factors involved with the "setting" problem is being studied. The results definitely indicate that the asphalt source, for any specific grade, is a most important factor. It should be again stressed that all of the paving on which these results were based was performed during a period of high day and night atmospheric temperatures. We have consistently found that "sensitive" asphalts may be used without trouble when atmospheric temperatures fall below approximately 80°F and this has been confirmed by the Corps of Engineers study (3). It is difficult to explain such findings even on the basis of the explanation offered in this paper or by those proposed by Schmidt and associates, (5, 6 & 7).

The results from the Cohesiograph test appear to provide a means of aiding in the controlling of the "setting" property of an asphalt paving mixture. We presently believe that requirements should be based on an original minimum reading and a minimum gain in length of break after the 24-hour heating period.

Unfortunately, the Cohesiograph test is primarily a research tool and presently there are problems in using the test for routine control. As previously noted, Fig. 2, there appears to be a fair relation between the original Cohesiograph reading and the absolute viscosity at the Cohesiograph test temperature. Further, we note from Figs. 4 & 5 that as the original Cohesiograph reading increases there is, in general, an increase in the gain in length of break between the original and 24-hour readings. Other investigators have maintained that the viscosity of the binder during compaction is of importance in connection with the "setting" problem and recently Traxler (8) has provided recommended minimum viscosity requirements at three different temperatures. These recommendations are based on the field "setting" performance of seven different 85-100 grade paving asphalts.

The absolute viscosity of the original asphalt at three different temperatures is compared with the original Cohesiograph reading in Fig. 6. The relation is quite good although the results are only based on California produced asphalts. Therefore, it may be possible to control the purchase of "sensitive" asphalts by the use of viscosity measurements at elevated temperatures, although further field and laboratory studies will be required.

In summary, independent field observations by three large consumer organizations in California, have clearly indicated the importance of the "setting" property of paving grade asphalts when construction is performed at high atmospheric temperatures. The Cohesiograph test appears to provide means of detecting "sensitive" asphalts, but the test is quite difficult to use for routine control, and preliminary studies indicate that satisfactory control may possibly be provided by viscosity tests at elevated temperatures.

There is a continuing need for further field and laboratory studies on this subject. Such studies should include field projects where it is possible to vary mix design, methods of compaction and sources of asphalt. It appears essential that such studies be performed during periods of high atmospheric temperatures and where it is necessary to route traffic over the pavement immediately after construction. There is also a continuing need for field test equipment that will provide a quantitative answer for the degree of "set" of a paving mixture.

We wish to state again that this problem has been encountered by the California Division of Highways for many years. During this period, we have never encountered a failure directly attributed to a slow "setting" paving mixture. The rolling operations may be difficult to perform and some "scuffing" under initial traffic may be encountered but the pavement "sets" in the first few weeks, and there is no evidence of any distress in terms of riding qualities. The problem is perhaps more serious in airfield pavements where densification is essential to avoid jet fuel spillage damage and in commercial and city street paving.

Durability

The most important property of any adhesive is its ability to act as an effective cementing agent during its service life. For an asphalt this includes a high degree of resistance to change in original properties during production of the paving mixture.

During the "mixing" process, paving grade asphalts are spread in thin films over the surface of the aggregate particles at elevated temperatures in order to properly coat the stone and thus form the paving mixture. During this process, the binder is subjected to conditions that may markedly change the original properties, and such changes, if drastic, may curtail the expected service life of the cementing agent. It is essential that adequate tests and specifications be provided to assure the engineer that the properties of the binder immediately following construction are satisfactory for future pavement performance.

After completion of construction and opening the pavement to traffic, the binder is subjected to a variety of traffic stresses imposed under different conditions of speed, loading and temperature. Further, the asphalt film is subjected to the elements and weathering may induce changes in the original engineering properties to the point where the binder is no longer effective.

The most pressing need is the development of suitable tests to measure changes in the engineering properties of the adhesive during mixing and subsequent pavement service life. All of our studies on this subject have been based on our belief that asphalt is subjected to two entirely different influences or conditions following manufacture. The first is during the mixing operation where aggregate temperatures are high, but the exposure time to such temperatures is very short. The second is during pavement service life where temperatures are relatively low, but the time extends over a period of many years. There are a number of reasons for believing that physical and chemical changes at elevated mixing temperatures are not in all respects, the same as those developed in a pavement at relatively low temperatures. It appears essential that tests be developed that will simulate each of these phases, as completely as possible, especially in terms of film thickness and temperature.

Our methods for studying the durability of paving grade asphalts are based on determining the changes in properties of thin films of asphalt at temperatures and test periods which closely simulate field conditions. The principal research method has been described in detail by Skog (9). In this method a two percent asphalt-Ottawa sand mix is used. Ottawa sand of No. 20 to No. 30 size was chosen in order to provide a medium for a statistically uniform film of asphalt for weathering studies, and with two percent of asphalt, a film thickness of about 5 to 7 microns is attained. The asphalt-Ottawa sand mixture is prepared in a laboratory mixer, and is then weathered in a special machine which has previously been described, (9). Essentially the unit weathers the sand-asphalt mixture in a semi-compacted state by subjecting the sample to infrared radiation which induces a constant mass temperature of 140°F with air maintained at 105°F flowing across the specimens. Radiant energy from the sun induces these temperature conditions in bituminous pavements throughout most of the United States during the summer months. Therefore, any acceleration produced in the oven is due only to the duration of time that the specimens are subjected to these temperatures. Changes in original properties of the binder as a result of "weathering" are determined by the Shot Abrasion test (9) and the Microviscometer technique.

Our first objective in this study was correlating the laboratory mixing unit with field mixing operations in order to place the binder in approximately the same physical state as typical of the pavement after construction. This was done by obtaining representative paving mixtures from a specific contract and determining the physical properties of the recovered asphalt. The average aggregate and asphalt temperatures during field mixing were also obtained. Using these temperatures, standard asphalt-Ottawa sand mixtures were made in the laboratory unit using various mixing times. The asphalt in the sand mixes was then recovered and physical tests performed. Preliminary studies indicated that a three minute mixing period in the laboratory using field temperatures would produce a satisfactory relation and a rather extensive program involving a large number of contracts was performed to check the validity of using the three minute period. The results for 29 separate contracts are shown in Fig. 7. The various projects involved different types of aggregates, mix types, asphalt grades and sources and field mixers. No attempt was made to control any of these variables. The only conditions compared were the same asphalt and average plant and aggregate temperatures as recorded on the plant report. Table A represents the results when typical softening points are compared on recovered asphalts from field and laboratory mixers. The change in temperature susceptibility is virtually the same and confirms the finding for the hardening rate as measured by the percentage of original penetration. We feel that the correlation is very good when one considers the uncontrolled variables. It is apparent that the source of the asphalt and the aggregate temperature are the two most important factors that are responsible for binder property changes during the field mixing process. We conclude that a satisfactory correlation has been attained between field and laboratory mixing and that changes in asphalt properties during field mixing may be duplicated in the laboratory mixer.

One may now approach the problem of asphalt durability in the pavement with some confidence, since it is now possible to prepare a mixture for weathering in which the binder closely simulates that discharged from a paving plant at equivalent aggregate and asphalt temperatures. Therefore, our next step was the calibration of our weathering machine in terms of pavement service time. In this case, we are not concerned with failure of the pavement, but simply to obtain an approximation of an equivalent unit of time between the weathering machine and pavements in California. It was realized, of course, that there are many variables influencing the rate of change in asphalt properties besides the single factor of temperature of weathering in the laboratory unit.

The procedure for weathering machine calibration may be outlined as follows, see Reference 9 for further details. Pavement cores from a specific project were obtained and the asphalt recovered by the Abson method. Normal physical tests were performed and the average abrasion loss of the recovered asphalt samples was determined by a solvent mixing method, Appendix B. It was necessary to develop this method since heating the recovered asphalt in order to prepare the Ottawa sand mixture would change the properties of the binder. Using an average sample of the original asphalt from the project and average field aggregate and asphalt temperatures, a standard 2% asphalt-Ottawa sand mixture was prepared in the laboratory mixer. The next step was to weather this mixture in the weathering machine under previously stated conditions. A normal weathering curve was then drawn, and the time in hours required to attain an abrasion loss comparable to the average core asphalt was determined. This length of time in hours in the machine is then equivalent to the service time in the pavement for the specific project. A typical determination is shown in Fig. 8.

The calibration study may be divided into two parts; namely, results obtained from a series of projects where only one or two asphalt sources were used on each job and results from the Zaca-Wigmore Project (10 & 11) where a series of asphalts were weathered under common conditions. Results from a series of projects involving one or two asphalt sources for each job are shown in Fig. 9. Results from the Zaca project are shown in Fig. 10. The curves from both series are compared in Fig. 11. The correlation between the two curves is very good and we may state with some confidence that exposure of 1000 hours in our weathering machine is approximately equal to five years of pavement service time for California conditions. The above noted relation apparently constitutes a fairly high rate of weathering. This has been partly confirmed by data made available to this department from the Virginia Test Road. We believe that 1000 hours of weathering in the laboratory unit is sufficient to evaluate an asphalt in terms of durability. This period, as previously mentioned, represents at least five years of pavement service life. Various investigators have consistently found that the most drastic changes in binder properties occur during the first thirty to thirty-six months of service life. Beyond this period there is a definite slow down in the rate of hardening which proceeds at a constant rate at least to the end of ten years service life.

After completion of the previously mentioned studies, the durability of a series of 40-50, 85-100 and 200-300 paving grade asphalts was determined by weathering for a period of 1000 hours. All Ottawa sand mixtures were prepared at a common mixing temperature of 325°F, the maximum aggregate temperature permitted in our field mixing operations. The use of this temperature subjected the asphalt to the most severe conditions encountered in field mixing. The following tests were performed on the Ottawa sand mixture after various periods of weathering to a total of 1000 hours.

1. Abrasion loss at 77°F
2. Loss of volatiles
3. Viscosity of the asphalt
4. Shear susceptibility

The Shell Development Research Group at Emeryville have developed a very satisfactory method for removal of asphalt from core slices or paving mixtures for viscosity determination. The asphalt is removed with benzene and large glass plates are coated with this solution. The benzene is removed by placing the plates in a box and flushing with nitrogen. The material is then scraped from a number of plates by means of a razor blade and viscosity specimens prepared.

The results of this study on the 85-100 grade series are shown in Table B and Figs. 12 and 13. The values shown include asphalts from different areas of the United States although the majority are from California production. The results clearly indicate the marked differences in the rate of weathering of asphalts from different sources as measured by both abrasion loss and viscosity tests. All of the asphalts complied in all respects with the presently used western States specification, which is more restrictive than general national requirements. Correlation studies indicate that 60 grams abrasion loss at 77°F is equivalent to approximately 10 penetration. We note that a number of these asphalts exceed this figure after approximately five years of service life in a California pavement.

It is very interesting to note, Table B, that the loss results during weathering are very low and in most cases a gain is shown after an initial period of slight loss. The results are based on carefully weighing specimens on an analytical balance after bringing to equilibrium in a desiccator. It is apparent that evaporation is not a factor in explaining the rate of change in abrasion loss and viscosity during the weathering period, in which the temperature never exceeds 140°F. The change must be caused by oxidation or other chemical modifications leading to a change in the rheological properties of the binder. The same results were obtained on studies of the 40-50 and 200-300 grades.

Another very interesting and important observation is the rate of change in "shear susceptibility" of various asphalts during weathering, Fig. 14. The shear index as shown on the ordinate of this figure is the tangent of the angle of the shear rate versus viscosity determined during performance of the viscosity test using the microviscometer. We note the marked differences in rate of change in shear index during weathering of different asphalts. It is most interesting to note that asphalt T which has the highest rate of change is of excellent durability as gaged by other tests, yet test pavements constructed with this asphalt failed after a short period of service life. The importance of "shear susceptibility" specifically the change in viscosity with shear rate in terms of pavement performance will be discussed in detail later in this paper.

Laboratory equipment has been constructed which will simulate the changes in asphalt properties during mixing and for at least five years of pavement service life. Various tests on the weathered binder clearly indicate the marked differences in durability of asphalts, from different sources. We believe that the above described equipment together with the correlation studies provides a means for characterizing the resistance to change of a paving grade asphalt during mixing and pavement service life.

The above described weathering method for measuring durability during mixing and pavement service life is too slow and costly for routine control testing. Therefore, our studies have included work on the development of test methods that will correlate with the research test and are practicable for routine control purposes.

Our first step in this direction was the development of a test for measuring resistance to change during the mixing operation. We are not completely satisfied with the presently used Thin Film test since neither the film thickness or the time in the oven are typical of field conditions. A new test called the Rolling Thin Film test is presented in detail in Appendix C and outlined briefly as follows: Glass bottles, of special design, containing 35 grams of asphalt are placed in a special rack in an oven. During the test period the rack is revolved at constant speed, causing the asphalt to flow in a thin film on the sides of the bottles. Based on previous observations of the color of thin films on exposure plates, we estimate the film thickness to be between 5-10 microns. Any possible volatiles which may collect are removed from each bottle, once in every revolution of the rack by a controlled jet of heated air. At the conclusion of the specified test period (say 1-1/4 hour) the bottles are removed and standard tests performed on the residue.

A critical factor in the test development was the determination of a time period that would place the asphalt in approximately the same physical state as found immediately after mixing. The procedure for determining a test period has been previously described, and on a series of asphalts the average time for the Rolling Thin Film test was found to be 73 minutes, Fig. 15. We have adopted a 75 minute test period. The suitability of this test period has been further confirmed by a correlation study with the calibrated Ottawa sand mixers. Tests were performed at two different test temperatures, 325°F and 375°F. The results are shown in Fig. 16. The correlation, appears satisfactory for both test temperatures and one may conclude that reaction rates, influenced by change in test temperature, are the same in both test methods.

Following development and completion of calibration studies, a study was performed to compare results of the Rolling Thin Film test and the Bureau of Public Roads (Lewis) Thin Film test. The comparison on a series of asphalts is shown in Fig. 17. Although there seems to be a trend, a close relation is not apparent and may be due to the differences in film thickness and length of test period.

The Rolling Thin Film test is a simple routine control method for predicting change in the properties of paving asphalts during the mixing process. The 75 minute test period has been established from actual field calibration, and asphalts subjected to this heat period should closely resemble in physical properties, the binder that is mixed in the field at an aggregate temperature equivalent to that used in the test.

Our next step was the use or modification of other published tests such as the Shell Microviscometer that might simulate the weathering during pavement service life. The objective was to match the changes in physical properties that occurred in the previously described weathering machine at the end of 1000 hours. Since thin films were used in the research weathering method, it was necessary to either increase the test temperature above 140°F or use oxygen under pressure in order to reduce the test time within the limits imposed for control during manufacture. We decided as a first step to raise the test temperature. The technique followed was the same as used in the determination of the Shell Aging Index (12, 13) except that the residue from the Rolling Thin Film test was used to prepare films on the glass plates. Further modifications were necessary since preliminary studies indicated that the recommended Shell test temperature was too high and the test period too short to provide a correlation with the 1000 hour readings from the research weathering unit. After a considerable number of trials a satisfactory relation was established. The modified Shell method is described in Appendix D and described briefly as follows: The residue from the Rolling Thin Film test is placed on 50mm x 50mm x 6mm glass plates and formed into films 20 microns thick using the Shell technique. The resulting 20-micron films are weathered in an oven maintained at 210°F for a period of 24 hours. The correlation between the results of this weathering period and those obtained after

1000 hours in the research weathering unit is shown in Fig. 18. The correlation appears quite satisfactory and leads to the conclusion that tests performed on the residue after this weathering period will approximate that found in the pavement after at least five years of service life. This study indicates that it is now possible to obtain a fairly good idea of the future durability of a paving asphalt, and that specification requirements may be written to control any potential change.

The tests that are performed on the residue after weathering should measure the rate of change in essential engineering properties of the binder. We readily admit that this is an involved problem and that field and laboratory studies must be continued. However, we do believe that there is now sufficient information to permit the writing of requirements to assure durability of an asphalt. This statement is based on the outstanding studies initiated by van der Poel (14) and further developed by others (15) (16), that the mechanical properties of an asphalt are dependent on the temperature and speed of loading. Although our tests on the residue do not fulfill the complete requirements of these investigations an initial attempt is made to follow these concepts.

The following discussion will be concerned with our studies on the residue from the 24-hour weathering period including an explanation of the possible significance of the proposed tests to indicate the durability of the binder. The residue is tested for viscosity at 77°F with a shear rate of 0.05 sec^{-1}, and using the "shear susceptibility" curve, the viscosity at 0.001 sec^{-1} is also determined. The Shell Development method (12)(17) is used for this determination, Appendix E. A new test, termed the micro-ductility test and developed by this department is also used to measure the change in ductile properties of the residue.

Although many technologists will be critical of the single determination of viscosity at 77°F after a pre-determined weathering period for indicating durability, a large number of reported field and laboratory investigations have clearly indicated that a critical hardness of a recovered asphalt, as measured at 77°F, does provide an index to possible failure of the binder to act as an effective cementing agent. A very interesting confirmation of this has been provided in the excellent studies by the Shell group on the recovered asphalts from the Zaca-Wigmore Project, (18). These investigators have provided data on viscosity measurements at various temperatures together with studies obtained with the micro-elastometer (19). The most significant measurement in respect to early pavement failure of three of the asphalts on this project is the change in the viscosity measurement at 77°F with a shear rate of 0.05 sec^{-1}.

Another very important factor has come to light since the publication of the studies by Doyle (20) in 1958. This very excellent investigation has, in our opinion, not received sufficient attention by asphalt technologists. We were very fortunate to be given a number of samples of asphalt that were used in the test roads and whose service performance is described in detail by P. C. Doyle. We have further confirmed these findings through the fortunate circumstance of incorporating an asphalt into the Zaca-Wigmore project that has failed in the same manner as certain of the Doyle asphalts.

The early failure of the Doyle asphalts "A" and "B" by cracking, pitting and raveling, in comparison with the regular production asphalts cannot be explained on the basis of temperature susceptibility only or by durability tests as measured by the abrasion loss or viscosity tests on weathered material. Both of these samples had outstanding resistance to change in properties as measured by these tests and had very low temperature susceptibilities. However, as shown in Fig. 19 these asphalts exhibited a marked change in "shear susceptibility" during weathering. This change will produce extremely high viscosities at low shear rates and ultimately could cause failure from contraction cracking. The cracking found in the test sections containing the "A" and "B" asphalts is definitely of this type. It is interesting to note, that the control asphalt C which remained in excellent condition has a much lower change in "shear susceptibility" and is virtually the same as another asphalt known to have excellent service performance.

Asphalt I placed in the Zaca-Wigmore Project (10) as an experimental material showed initial pitting and slight raveling within two years after construction. The difference in surface appearance between this pavement and the adjacent section constructed with a normal production binder was very striking. Since the surfacing for both of these test sections was laid on the same day under identical construction conditions the only answer to this difference is in the nature of the asphalt. Temperature changes on this project are very mild and typical contraction cracking in both lanes did not appear until after six years of service life, but had increased quite markedly after seven years, Fig. 20. The pavement deflections for this section are consistently low and there is little evidence of "chicken" wire cracking.

We have also been furnished a foreign asphalt that closely resembles the three previously discussed materials in its physical property changes during weathering. Pavements laid with this binder in the desert regions have failed by contraction cracking within two or three years after construction.

All of these binders have certain common test properties. They all exhibit a marked decrease in ductility after loss-on-heat tests and a marked gain in "shear susceptibility" by both methods of weathering described in this paper. R. S. Winniford (21) has shown that these properties indicate a highly gelled asphalt.

The viscosity at different rates of shear after 1000 hours of weathering are compared with satisfactory binders in Fig. 21. Although the field information is somewhat meager it is apparent that the results are significant. We are aware that the change in "shear susceptibility" with temperature is a variable factor for different asphalts and that some investigators believe that 77°F is not an appropriate temperature. However, the exact field conditions in terms of temperature ranges that induce failures are virtually unknown.

The above mentioned field failures indicate that it is necessary to control "shear susceptibility" in any future asphalt specification. There is reason to believe that the different opinions expressed on the service behavior of partially b paving asphalts may be partly explained by the change in "shear susceptibility" during weathering, leading to extremely high viscosities at a slow speed loading. It should be again stressed that the "shear susceptibility" factor cannot be controlled on the original sample or even after mixing. This has been well illustrated in Fig. 14. Further, the problem is complicated by the fact that an asphalt having a low "shear susceptibility" during weathering may harden at such a high rate that its viscosity at slow speed loading may become critical. Therefore, it is essential to determine all of these property changes on material weathered for some specified time that simulates a definite period of pavement service life.

The third measurement on the residue from the routine control weathering test is concerned with a determination of ductility. One of the most debated methods for controlling asphalt quality is the importance and significance of the ductility test. There is little reason to doubt that the original ductility, as measured under present standard conditions, has little or no meaning in terms of future pavement performance. This is well illustrated in the case of California produced asphalts where binders having a wide range in service life durability all show a 100+ ductility. However, change in ductility during service life appears to have definite significance. Results reported in the literature (22, 23, 24, 25, 26) from various field and laboratory investigations clearly indicate that a decrease in ductile properties during weathering, as measured by the standard test, is related to failure of the pavement.

R. G. Clark (25) in a recent report states: "It is our considered opinion, however, based on the analysis of many pavements, that the ductility of an asphalt, especially after it has been incorporated into the pavement is of prime importance in determining the quality of a bituminous structure". There appears to be increasing evidence that the ductility test on asphalts recovered from the pavement during service life is an important method for judging service performance. Exactly what property is being measured is difficult to decide, but the rate of change in test results is significant when coupled with the rate of change in penetration. The fact that the significance of ductility only becomes apparent during service life presents a problem in measurement with the standard apparatus, since generally only a small amount of material is available after a laboratory weathering period. A test has recently been developed which provides a method of determining ductility on a very small amount (0.05 gram is sufficient) of weathered material. The method is described in Appendix F. Results for various asphalts after 24 hours of weathering at 210°F are shown in Tables C and D. It is interesting to note, Table D, that the ductility results for unsatisfactory asphalts on the Zaca-Wigmore project are very low.

Another factor that may alter the durability of a paving grade asphalt is syneresis or oil exudation tendencies. This problem has mainly concerned individuals connected with the manufacture of roofing and laminated paper where undesirable staining may occur. The tendency of an asphalt to exhibit oil exudation is most commonly found in the highly air refined type although G. L. Oliensis (27) states: "Even lightly oxidized asphalts and fairly stable blends of bitumens not to mention some straight reduced petroleum residuals may develop the same tendency, though frequently in those cases this trend is very slight." However, asphalts produced to conform to new requirements involving "set" and durability may require partial "blowing" or special synthesis. This may lead to an increase in the tendency towards oil exudation. The preferential adsorption of the exuded oils by certain aggregate types could produce a hard and possibly brittle adhesive. The need for control of syneresis even in paving grade asphalts is shown by the Doyle sample "A" which has a very high degree of syneresis, but meets certain other durability requirements.

Studies on this phase of durability were performed by using the present ASTM D1328 method for measuring oil exudation tendencies. Modifications in the test variables of temperature, pressure and time were studied in applying the method to paving grade asphalts. The test temperature was set at 140°F, the average maximum pavement temperature, and the pressure at 50 psi. The time period for best differentiation was determined by observing the stain number after 24, 48, 72 and 120 hours on a series of 40-50, 85-100 and 200-300 grade paving asphalts. The results of this study for the 85-100 grade are shown in Fig. 22. The results indicate that a 120-hour period is most desirable for differentiation. Fig. 23 presents the results on a number of 40-50, 85-100 and 200-300 grade asphalts after the 120-hour test. It is interesting to note that some 40-50 asphalts have higher stain numbers than 200-300 grade binders from other sources. The results on the 200-300 series are those obtained from the Zaca-Wigmore project. The highest stain numbers are shown by two asphalts which have failed. The mechanism of failure cannot be assigned to oil exudation tendencies, however, both of these asphalts exhibited a high rate of change in "shear susceptibility" during weathering and the stain number provides a secondary indication of this possible property change. Although the oil exudation property of paving grade asphalts cannot be considered as an important factor in durability, the test does provide a method of attaining more balanced engineering properties, and should be utilized in any new specification.

Resistance to Water Action

It is well known that the properties of the aggregate surface are of prime importance in the resistance of a pavement to action of water. However, the asphalt may affect the overall resistance as shown in Table E where results obtained with the Quantitative Dye technique are shown, (28). There is a difference in the stripping resistance of a paving mixture containing a "sensitive" aggregate when different asphalt sources of the same grade are compared. There appears to be a need for direct evaluation of asphalt properties that are concerned with the water action problem.

Studies performed by this department with a modification of the test developed by Thelen (29) have not been successful. Further studies on this problem are required.

SPECIFICATIONS

The need for new specifications for paving grade asphalts involving tests that will provide better criteria for the four basic properties noted in this paper has been recognized for a number of years. The major portion of this paper has been devoted to a description of our studies on the development of tests for "setting" and durability properties. Methods of measurement for the consistency property appear to be quite satisfactory except for low temperature measurements, but there is need for continued studies on the water action property. We believe that there is now available, sufficient information on test development and field performance so that a new specification for paving asphalts may be proposed. Further, there are indications that a further reduction in grades of asphalt may be in order if asphalts meeting the new requirements are produced.

Grades

During the second Pacific Coast Conference on Asphalt Specifications in San Francisco, February, 1957, the six western States agreed to accept five grades of paving asphalt; namely, 40-50, 60-70, 85-100, 120-150 and 200-300. Since this time the trend in California has been to the use of the 85-100 grade for almost all of our paving work. The use of the 200-300 grade has virtually ceased and the consumption of 120-150 has markedly fallen. There is every indication that the 85-100 grade has been successfully used in all areas of the State under varied paving and atmospheric conditions.

We now believe that satisfactory highway paving under almost any atmospheric condition or mix design may be accomplished with three grades of paving asphalt; namely, 50-60, 85-100 and 175-225.

The 85-100 grade is proposed as the most common paving material for all forms of highway construction. At the present time, there may be some question whether it is possible to produce a 40-50 grade that will comply with our proposed requirements for durability. On the other hand, heavy traffic demands may require the future use of asphalts of lower consistency than the 85-100 grade. We are, therefore, proposing a 50-60 grade for this purpose.

There appears to be a need for a relatively soft grade, mainly for late fall and early spring paving. The present 200-300 grade has too broad a range and the abandonment of this grade in California paving during the past five years indicates a need for revision. Therefore, a 175-225 grade is proposed which will permit closer control and more restrictive requirements for use as a paving material.

Specification Tests and Requirements

A tentative specification is shown in Table F. The proposed requirements should be considered only as tentative and subject to further field and laboratory study. They are somewhat restrictive and the presently produced 85-100 grade asphalts that we have tested will not comply in all respects with the proposed requirements. This does not imply that it will be impossible to produce asphalts to comply with these specifications. It simply means that most of the requirements have not appeared in previous specifications and present production does not provide materials that have been controlled by methods proposed in these requirements. The most important factor that has been constantly considered in the writing of these requirements is to attain a balance between the various desired property values. The great importance of this factor is shown by the results reported by Doyle, and previously discussed in this paper. In this case highly durable materials of exceptionally low temperature susceptibility and anti-brittleness qualities failed after a short period of service life. Apparently, in order to produce these exceptional values for one set of properties, it was necessary to detrimentally influence other important factors in binder performance and failure resulted. On the other hand, the normal control asphalts that had previously provided satisfactory pavement performance had good values in all of the tests and provided a balanced set of engineering properties.

The specification requirements are considered in some detail below:

Flash

The flash test in these specifications should be mainly considered in terms of the safety factor since the required durability tests for mixing and service life are restrictive. Based on our studies, it appears that asphalts complying with the durability requirements will also have a high flash. The test may have value by insuring more uniform production from various sources.

Original Consistency

We have continued the present method of defining grade by use of the standard Penetration test at 77°F.

This method seems satisfactory for the present since any change to absolute viscosity at other temperatures would require a rather extensive program to familiarize field engineers with new terms. It seems far more important to attain asphalts having satisfactory properties during mixing, placement and service life.

"Setting" Requirements

As mentioned previously, we do not presently believe that the Cohesiograph test is adaptable to routine control. Therefore, we propose to attempt controlling the "setting" property by viscosity requirements at 140° and 225°F. The minimum values are determined from the required minimum Cohesiograph readings. Actually, it would be preferable to control the viscosity requirements on the basis of tests on the Rolling Thin Film residue, but this is not possible, at present, since the minimum requirements for the penetration on the residue may prevent the attainment of the required viscosities at elevated temperature. The establishment of minimum viscosities at 140° and 225°F for the purpose of controlling the "setting" property, also fixes the temperature susceptibility of the binder at elevated temperatures, if a secondary control viscosity at 325°F is established. The maximum stated viscosity at 325°F assures the engineer that the binder will fall in the range of 75-150 seconds Saybold Furol viscosity prior to the maximum aggregate temperature (325°F) permitted by our specifications. Although the slopes of temperature - viscosity relationships between 40 - 77°F and 77° - 325°F have been found to be somewhat different, the differences are not great enough to require a maximum viscosity at 40°F and we have, therefore, not specified a low temperature viscosity requirement. We, however, feel that this requirement should be specified after further study of various methods of measurement. Although the experimental studies indicate the definite possibility of adequately controlling "set" by elevated viscosity measurements, we are still including a Cohesiograph requirement in our tentative specifications. Compliance with these requirements will be performed in the consumer laboratory on occasional samples drawn from current production as desired by the consumer. Further, there is the possible production of asphalts having a definitely different temperature susceptibility above 140°F from that found below this temperature. This would be an ideal material since mixing, placement and compaction could be performed at relatively low temperatures, with the added advantage of low temperature susceptibility during service life. With this in mind, a special note #1 is attached to the tentative specification. The continued use of the Cohesiograph test in these tentative specifications will depend on future field experience with asphalts complying with the new requirements.

Durability

The most important aspect of these specifications is the control of durability during mixing and pavement service life. We have substituted the Rolling Thin Film Test for the present Bureau of Public Roads Thin Film test on the premise that such tests should be performed in a true thin film state and for a time period that has been correlated with field mixer performance. We have also abandoned the presently used method of reporting the percentage of original penetration and simply specify the actual minimum penetration on the residue. This provides a better understood figure for a requirement of this type. The proposed requirements for retained penetration are more restrictive than those presently used in the western States specification on the belief that asphalts must meet at least this minimum if the service life requirements are to be met. These values, however, may be modified when materials meeting the service life requirements are available for study.

In order to produce satisfactory compaction during the late fall and early spring, it may be necessary to raise plant aggregate temperatures above the maximum presently found in most specifications. We have, therefore, proposed additional requirements for the bituminous binders used under these conditions since it is known that some asphalts are drastically hardened by temperatures in excess of 325°F.

The values for the viscosities at two different shear rates and the micro-ductility on the residue after 24 hours at 210°F are based on the available field and laboratory investigations of this and other organizations. It is well known that numerous investigators have determined that when the recovered asphalt penetration at 77°F is around 20 and the ductility has fallen to a low value, the binder is in a critical state and failure may quickly result in areas of relatively high pavement deflection. It seems essential to limit the viscosity at a shear rate of 0.05 sec^{-1} to some value that is close to this penetration value. We believe that asphalts that do not exceed 20 megapoises viscosity at 77°F, shear rate 0.05 sec^{-1} will provide a very satisfactory binder during service life, if the ductility requirements are also met. Further, we hope to control contraction cracking by limiting the viscosity at 0.001 sec^{-1} shear rate to 60. This value is based on available field evidence as presented in Fig. 21. The values are identical for all three proposed grades of paving asphalt. We assume that these measurements provide to some degree an evaluation of the critical state of the binder in terms of its ability to act as an effective cementing agent.

Syneresis or the tendency of the asphalt to exude oils is controlled by the stain test. The exact significance of this requirement is difficult to decide at present, but in the presence of certain types of aggregates excessive oil exudation could lead to early hardening of the binder film. Finally, all asphalt specifications should contain a purity requirement and the Carbon Tetrachloride test is adequate for this purpose.

In summary, a new specification for paving grade asphalts involving a minimum number of grades has been proposed. The primary purpose of the specification is to provide a <u>balanced</u> set of engineering properties together with excellent durability. There is still a need for more satisfactory control tests for "setting" and low temperature properties, and the development of a method for controlling the surface affinity of the binder when the pavement is subjected to water action.

SUMMARY AND CONCLUSIONS

Asphalt quality for paving purposes may be evaluated by four basic properties; namely, consistency, "setting", durability and resistance to water action. Analyses of failed pavements in which the binder is directly concerned indicate that an evaluation of these four properties with proper tests and requirements will produce a satisfactory binder for highway construction.

Field observations by three consumer organizations in California, California Division of Highways, Corps of Engineers, Sacramento District, and Industrial Asphalt Company, indicate that four field factors influence the "setting" properties of asphalt concrete; namely, atmospheric temperatures during and immediately after construction, asphalt source, mix design, and traffic requirements during and immediately after construction. The most important variables appear to be the atmospheric temperatures and source of the asphalt. "Sensitive" asphalts become satisfactory when atmospheric temperatures fall below approximately 80°F.

The Cohesiograph test provides a test method for determining if an asphalt will contribute to slow "setting" characteristics of a paving mixture, and requirements are proposed for controlling this property. The test does not appear, at present, to be adaptable for control purposes and viscosities at elevated temperatures are being substituted for routine control.

A research test for determining the durability of paving grade asphalts during mixing and pavement service life has been developed and correlated with field performance. Various tests during laboratory weathering indicate that asphalts complying with presently used specifications vary widely in their resistance to change under temperatures that closely simulate field conditions. Tests for routine control have been correlated with the research weathering device in terms of time equivalent to at least five years of pavement service life.

A tentative specification has been presented. The requirements are based on existing and newly developed tests that provide a better indication of the required properties of a paving grade asphalt. An attempt has been made to balance the various requirements and attain a product of uniform engineering properties from various sources. We believe that this is the first attempt to write a specification wherein tests developed by research groups are used for control of production. The only way that continuing research on paving grade asphalts will be of value to consumer organizations is the ability of the engineer to use such results for specification purposes.

There are many problems that remain unsolved in the field of asphalt technology. An understanding of the fundamental nature of asphalt and the response of the binder to variable conditions of stress during service life still requires the continued combined efforts of User-Producer groups.

ACKNOWLEDGMENTS

The writers wish to acknowledge the complete cooperation of the asphalt producers of California, and other organizations in furnishing samples and other information essential for the reported studies.

Special acknowledgment is due Messrs. Glenn Kemp, James Kreiss, Donald Vert, Nelson Predoehl and Gene Stucky, who performed many of the laboratory studies.

REFERENCES

1. F. N. Hveem
"Quality Tests for Asphalts - A Progress Report"
Association of Asphalt Paving Technologists, Vol. 15, p. 111, 1943

2. R. L. Griffin, T. K. Miles, C. J. Penther and W. C. Simpson
"Sliding Plate Microviscometer for Rapid Measurement of Asphalt Viscosity in Absolute Units"
ASTM Special Technical Publication No. 212

3. D. D. Fenton
"Field Experience and Laboratory Study of 'Slow Setting' Paving Grade Asphalt in Airfield Pavement Construction"
ASTM Vol. 58, p. 1306, 1958

4. E. J. Woodward, Jr.
"Developing HLR - A Penetration Type Asphalt Made to Withstand A Static Load at Changing Temperatures"
National Bituminous Concrete Association, Houston, Texas, January 28, 1961

5. R. J. Schmidt, W. J. Kari, H. C. Bower and T. C. Hein
"Behavior of Hot Asphaltic Concrete Under Steel-Wheel Rollers"
HRB Bulletin No. 251, 1960

6. L. E. Santucci and R. J. Schmidt
"Setting Rate of Asphalt Concrete"
HRB Annual Meeting, 1962

7. R. J. Schmidt and L. E. Santucci
"Influence of Asphalt Type on Pavement Setting Rate"
HRB Annual Meeting, 1962

8. R. N. Traxler
"Current Problems in Bituminous Technology"
Texas Engineering Experiment Station News, Vol. 13, No. 1, p. 3, 1962

9. J. B. Skog
"The Operation, Control and Application of the Infrared Weathering Machine-California Design"
ASTM - Special Technical Publication No. 212, 1957

10. F. N. Hveem, E. Zube and J. Skog
"Progress Report on the Zaca-Wigmore Experimental Asphalt Test Project"
ASTM Special Technical Publication No. 277, 1959

11. E. Zube
"Asphalt Test"
California Highways and Public Works, May-June 1961

12. R. L. Griffin, T. K. Miles and C. J. Penther
"Microfilm Durability Test for Asphalts"
Proceedings, Association of Asphalt Paving Technologists, Vol. 24, p. 31, 1955

13. D. F. Fink and J. J. Heithaus
"The Precision of Measurements With the Sliding Plate Microviscometer, Appendix I."
ASTM Special Technical Publication No. 309, 1961

14. C. van der Poel
"A General System Describing the Viscoelastic Properties of Bitumens and Its Relation to Routine Test Data"
Journal of Applied Chemistry, Vol. 4, p. 221, 1954

15. J. C. Brodnyan
"Use of Rheological and Other Data in Asphalt Engineering Problems"
Highway Research Board Bulletin 192, p. 1, 1958

16. S. H. Kühn and P. J. Rigden
"Measurement of Visco-Elastic Properties of Bitumens Under Dynamic Loading"
Proceedings, Highway Research Board, Vol. 38, p. 431, 1959

17. D. F. Fink and R. L. Griffin
"Measurement of the Consistency of Paving Cements at 140F With the Sliding Plate Microviscometer"
ASTM Special Technical Publication No. 309, 1961

18. Discussion at the Fifteenth Annual Meeting of the Triaxial Institute, Klamath Falls, Oregon, March 1962, by G. A. McLaren, Shell Oil Company

19. J. W. A. Labout
"The Microelastometer, a Microdevice for Measuring the Modulus of 'Stiffness' of Small Samples of Thermoplastic Materials"
Rheologica Acta, I, 186-190
(Ergänzungshefte zur Kolloid-Zeitschrift) (August 1958)

20. P. C. Doyle
"Cracking Characteristic of Asphalt Cement"
Proceedings of the Association of Asphalt Paving
Technologists, Vol. 27, p. 581, 1958

21. R. S. Winniford
"The Influence of Asphalt Composition on Its Rheology"
ASTM Meeting, Atlantic City, June 27, 1960

22. R. Vokac
"Correlation of Laboratory Tests of Bituminous Mixtures
With Service Behavior"
Proceedings of the Montana National Bituminous Conference
Sept. 1939, p. 226

23. A Study of Bituminous Concrete Pavements in Ohio
Ohio Department of Highways and the Bureau of Public Roads
Public Roads, August 1941

24. J. T. Pauls and J. Y. Welborn
"Studies of Hardening Properties of Asphaltic Materials"
Public Roads, August 1953

25. R. G. Clark
"Practical Results of Asphalt Hardening on Pavement Life"
Proceedings of the Association of Asphalt Paving
Technologists, Vol. 27, p. 196, 1958

26. J. F. Goode and E. P. Owings
"A Laboratory-Field Study of Hot Asphaltic Concrete
Wearing Course Mixtures"
ASTM Special Technical Publication No. 309, 1961

27. G. L. Oliensis
"Exudation and Allied Reactions Between Bitumens"
Forest Publishing Co., Forest Park, Illinois, 1949

28. J. Skog and E. Zube
"New Test Methods for Studying the Effect of Water
Action on Bituminous Mixtures"
Association of Asphalt Paving Technologists Meeting,
San Francisco, February 1963

29. E. Thelen
"Surface Energy and Adhesion Properties in Asphalt-
Aggregate Systems"
Highway Research Board Bulletin 192, 1958

TABLE A

Laboratory Mixer Calibration
Comparison of Softening Points

Contract	Asphalt Source	Asphalt Grade	Softening Point of Recovered Asphalt	
			Field Mixer	Laboratory Mixer
1	C	85-100	(49)* 130	(51)* 132
2	C	150-200	(122) 107	(123) 108
3	C	150-200	(114) 104	(114) 105
4	C	200-300	(155) 102	(148) 105
5	B	120-150	(62) 119	(74) 120
6	D	85-100	(66) 115	(63) 117
7	F	120-150	(62) 126	(67) 124
8	F	150-200	(72) 122	(71) 124
9	A	120-150	(72) 117	(78) 118

*Recovered Penetration

TABLE B

Volatile Loss During Weathering of Various 85-100 Grade Paving Asphalts.

Asphalt Source	Total Max. Loss		Total Loss or Gain After 1000 Hours Weathering
	Hours of Weathering	Loss %	
A	-	-	-0.002%
B	567	-0.002	+0.001
D	44	-0.001	+0.013
F	-	-	-0.004
G	-	-	-0.002
H	-	-	-0.002
I	258	-0.002	+0.004
J	90	-0.001	+0.009
K	41	-0.001	+0.007
L	232	-0.001	+0.003
M	-	-	-0.002
N	45	-0.001	+0.013
O	-	-	-0.004
P	44	-0.004	+0.006
Q	210	-0.004	+0.006
R	42	-0.002	+0.011
S	41	-0.002	+0.012
T	-	-	-0.024
U	-	-	-0.001
V	210	-0.002	+0.010
W	233	-0.005	+0.009
X	44	-0.006	+0.012

TABLE C

Ductility Results After Durability Test for Various 40-50 and 85-100 Grade Paving Asphalts

Asphalt Source	Asphalt Grade	Ductility After Durability Test 1/2 cm/min. at 77°F (cm.)
A	40-50	0
B	"	0.2
D	"	0.3
F	"	0.1
I	"	0
S	"	0.2
A	85-100	0.3
B	"	0.8
D	"	7.5
F	"	0.2
I	"	1.3
O	"	0.2
Y	"	0.1
W	"	6.0
X	"	0.8
A-1	"	2.3
A-2	"	0.4
A-3	"	0.2
B-1	"	0.9
B-2	"	0.4
B-3	"	0.7

TABLE D

Ductility Results After Durability Test for Zaca-Wigmore Project Asphalts

Asphalt Source	Paving Period	Asphalt Grade	Ductility After Durability Test 1/2 cm/min. at 77°F (cm.)
A	I	200-300	8.8
B-1	IA	"	1.3
C-1	IA	"	5.9
C	I	"	5.2
D	"	"	6.1
E	"	"	0
F	"	"	0.5
G	"	"	0.2
H	"	"	2.5
J	"	"	3.9
A-2	II	200-300	6.1
B-2	"	"	0.5
C-2	"	"	5.2
D-2	"	"	8.0
E-2	"	"	0
G-2	"	"	0.2
H-2	"	"	2.9
I-2	"	"	0.3

TABLE E

Influence of Asphalt Source on Percentage of Stripping as Measured by the Dye Technique

Aggregate Source	Asphalt Source	Grade	Curing Time Hrs. at 140°F	Film Stripping Test			Dye Test % Stripping
				Method	Test Temp. °F	Test Time Min.	
III	1	85-100	15	Calif. 302-B	77	30	35
"	2	"	"	"	"	"	45
"	3	"	"	"	"	"	53
"	4	"	"	"	"	"	65
"	5	"	"	"	"	"	71

TABLE F

TENTATIVE SPECIFICATIONS FOR PAVING GRADE ASPHALTS

Specification Designation	Test Method	Grade 50-60	Grade 85-100	Grade 175-225
Flash Point PMCT °F Minimum	AASHO T73	500	475	450
Penetration of Original Sample at 77°F	AASHO T49	50-60	85-100	175-225
Stain Number of Orig. Sample Max. After 120 Hrs. - 140°F - 50#/sq.in.	ASTM D1328-58T	10	10	10
Viscosity, cs on Orig. Sample	ASTM D445			
140°F, Minimum		3.7×10^5	2.2×10^5	1.1×10^5
225°F, Minimum(1)		2500	1800	1100
325°F, Maximum		200	200	200
Cohesiograph(2) Reading	Calif. Test Method No. 350			
Orig. Minimum, Inches		0.95	0.80	0.65
Gain 0-24 Hrs. Minimum, Inches		0.10	0.08	0.06
Rolling Thin Film Test at 325°F, 75 Min.	Calif. Test Method No. 346			
Pen. Residue 77°F, Minimum	AASHO T49	40	55	125
Duct. Residue 77°F, 5 cm./min., Minimum, cm.	AASHO T51	75	75	75
Durability Test(3)	Calif. Test Method No. 347			
Viscosity of Residue After Durability Test.				
Megapoises at 77°F Shear Rate - 0.05 sec^{-1}, Maximum	Calif. Test Method No. 348	20	20	20
Megapoises at 77°F Shear Rate - 0.001 sec^{-1}, Maximum		60	60	60
Micro Ductility of Residue After Durability Test. 77°F, 1/2 cm./min., Minimum, mm.	Calif. Test Method No. 349	10	10	10
Solubility in CCl_4 Original Sample % Min.	AASHO T45	99	99	99

TABLE F (Cont'd)

Tentative Specifications for
Paving Grade Asphalts

Additional requirements for all asphalts delivered to any contract after October 1 and prior to April 1.

Specification Designation	Test Method	Grade 50-60	Grade 85-100	Grade 175-225
Rolling Thin Film Test at 375°F, 75 Min.	Calif. Test Method No. 346			
Pen. Residue, 77°F, Minimum	AASHO T49	35	45	100
Duct. Residue, 77°F, 5 cm./min., Minimum, cm.	AASHO T51	60	60	60

Notes

1. If the viscosity at 225°F is below the required minimum, but the asphalt complies with the requirements for the viscosity at 140°F and the Cohesiograph test, the material shall be acceptable.

2. The manufacturer shall furnish one gallon samples prior to initial shipments of new production, and as requested thereafter for the Cohesiograph test. The test shall be performed in the Materials and Research Department of the California Division of Highways.

3. Durability test to be performed on the residue from the 325°F Rolling Thin Film Test.

FIGURE 2

COMPARISON OF ROLLING THIN FILM RESIDUE VISCOSITY AND ORIGINAL COHESIOGRAPH READING

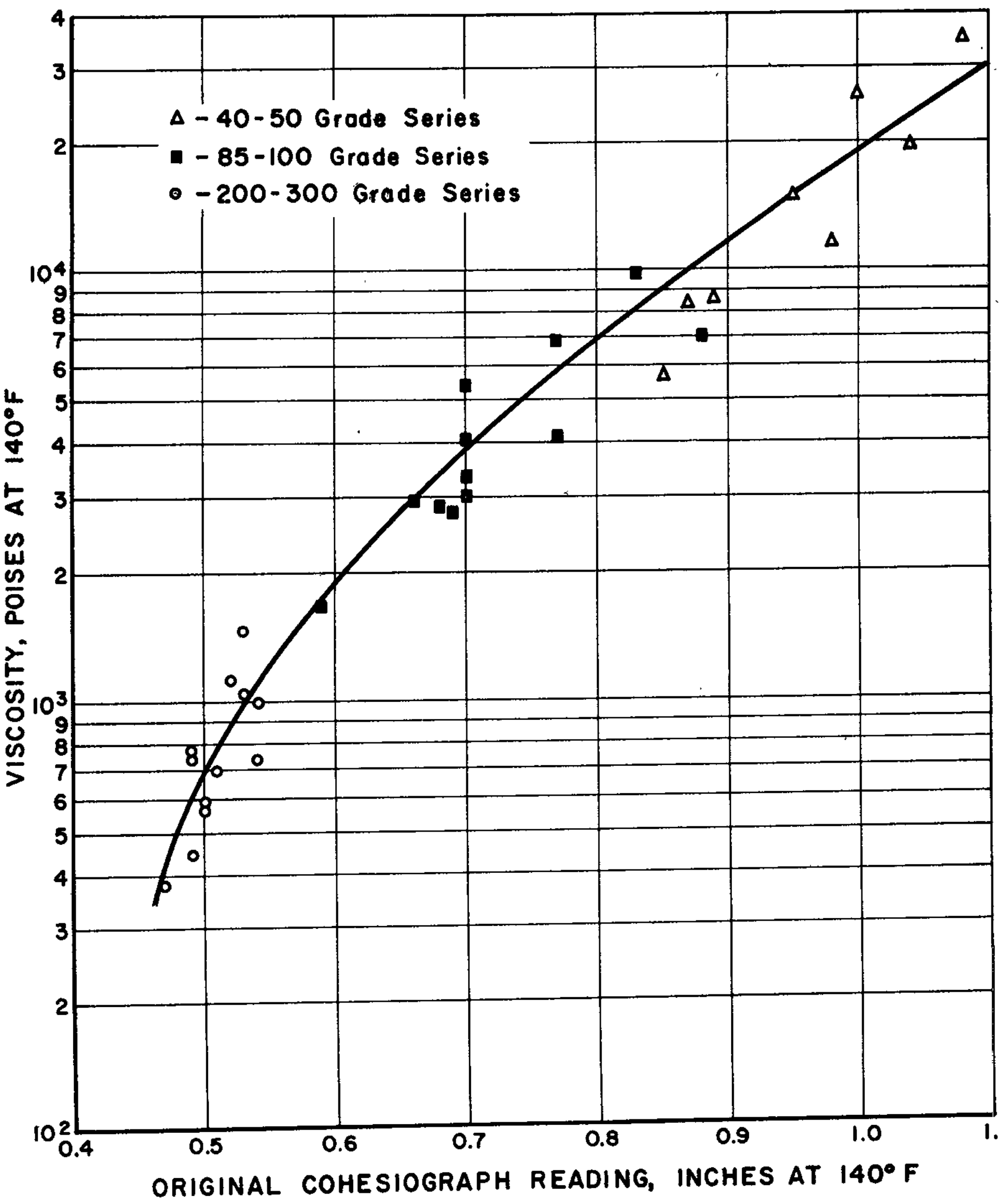


FIGURE 3

COHESIOGRAPH RESULTS ON VARIOUS 85-100 GRADE PAVING ASPHALTS

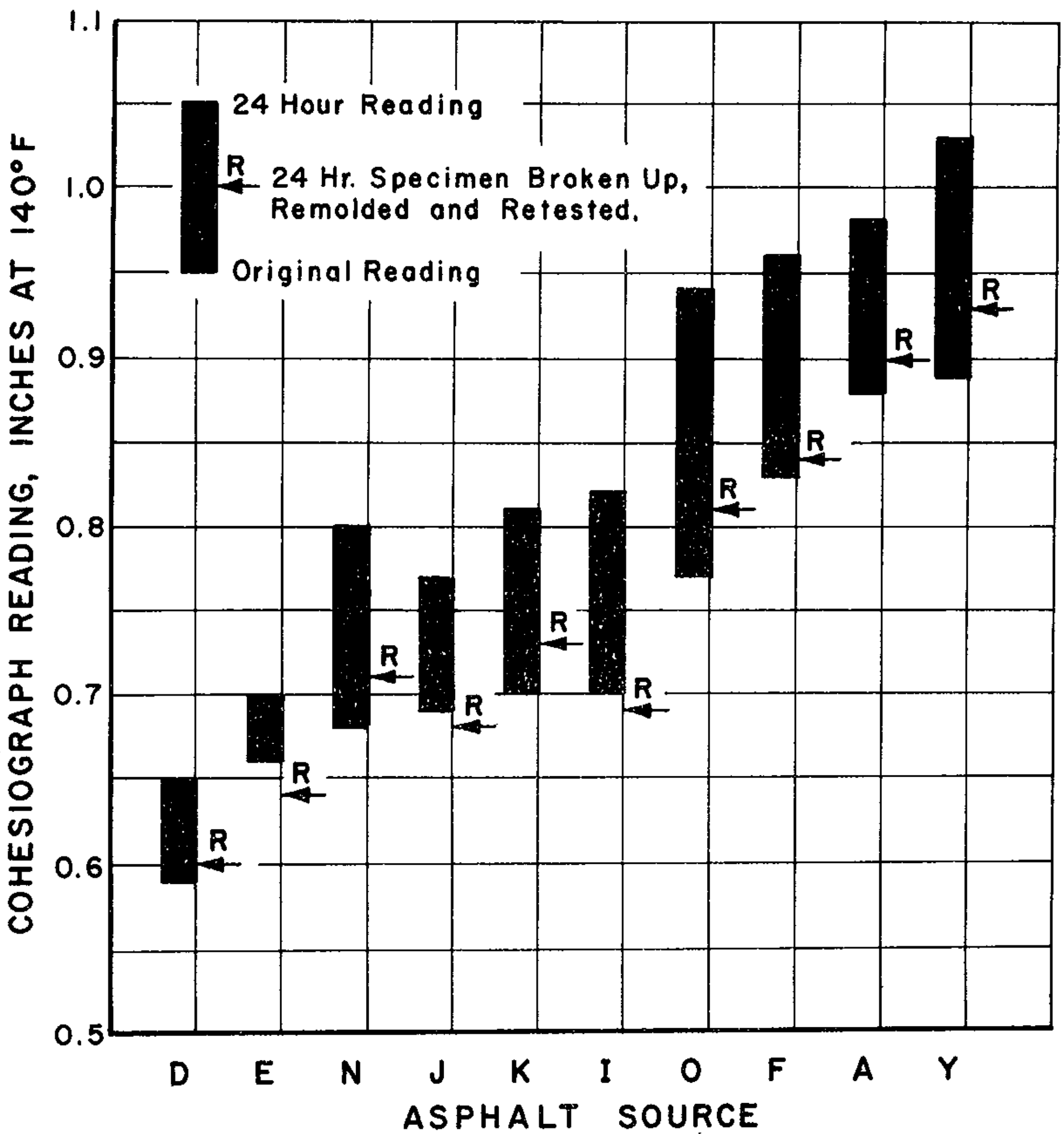


FIGURE 4

FIELD "SETTING" PERFORMANCE – COHESIOGRAPH CORRELATION STUDY ON 85-100 GRADE PAVING ASPHALTS

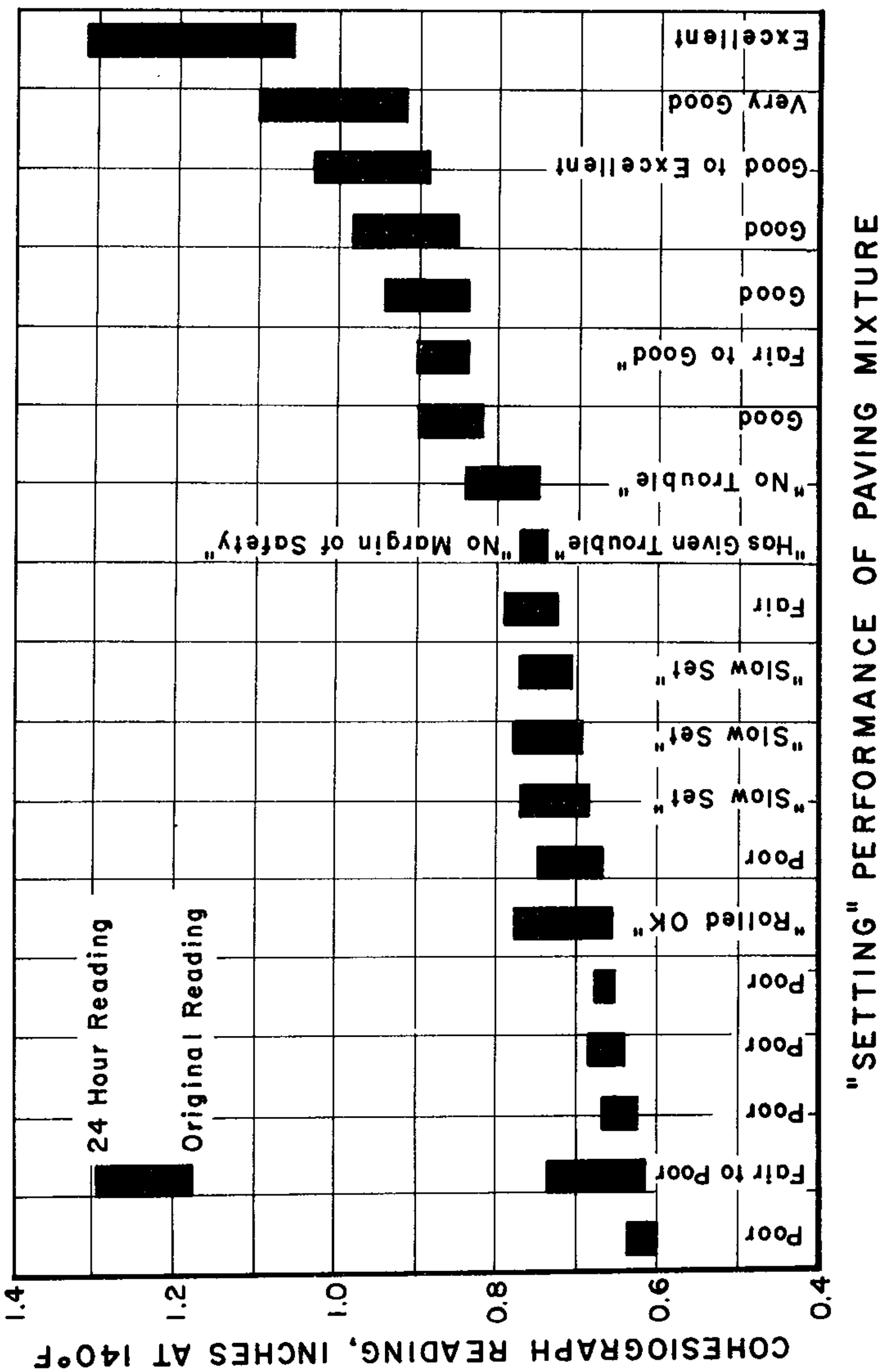


FIGURE 5

FIELD "SETTING" PERFORMANCE
COHESIOGRAPH CORRELATION STUDY
ON 120-150 AND 200-300 GRADE PAVING ASPHALTS

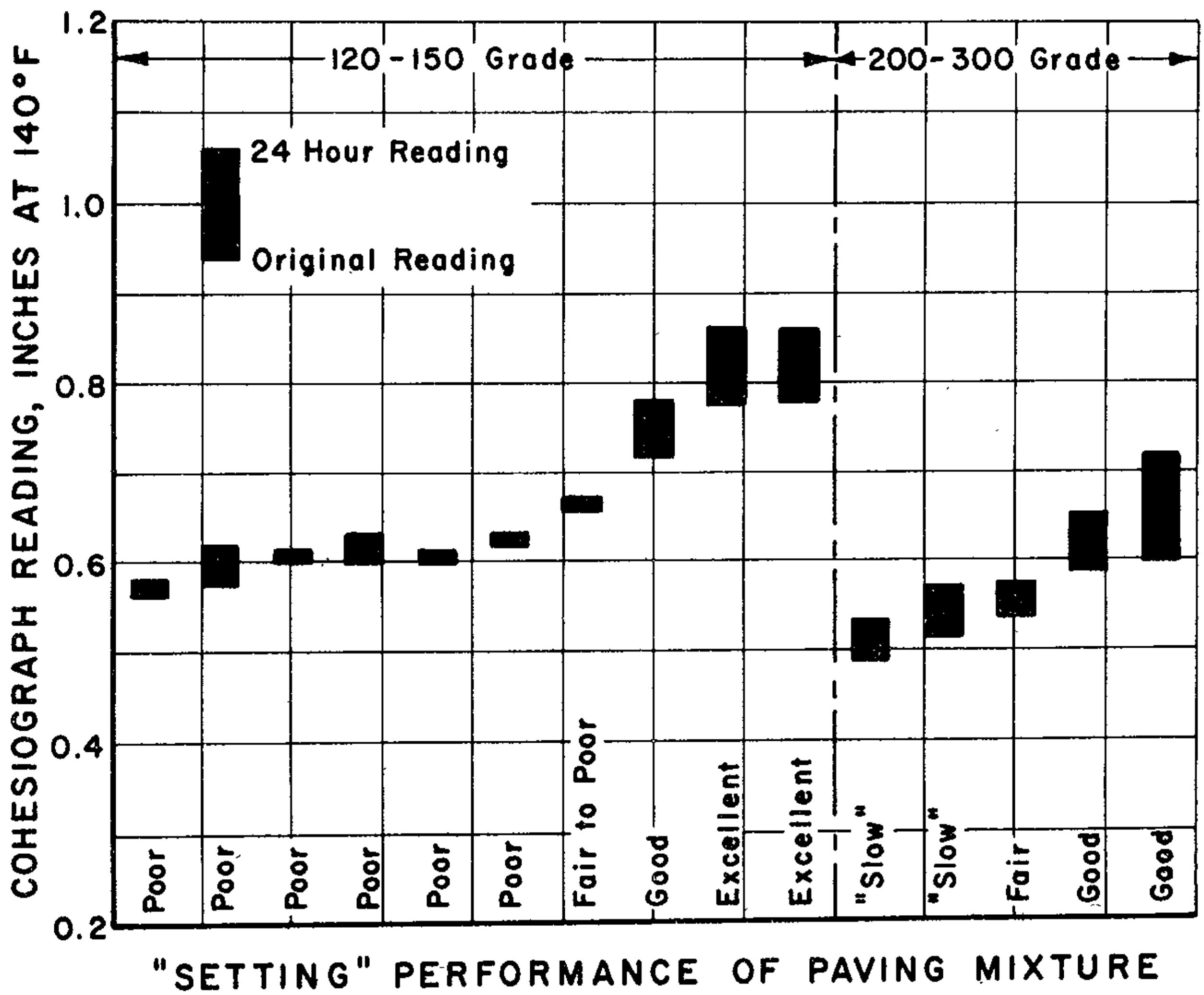


FIGURE 6

RELATIONSHIP BETWEEN VISCOSITY OF ORIGINAL ASPHALT AND ORIGINAL COHESIOGRAPH READING

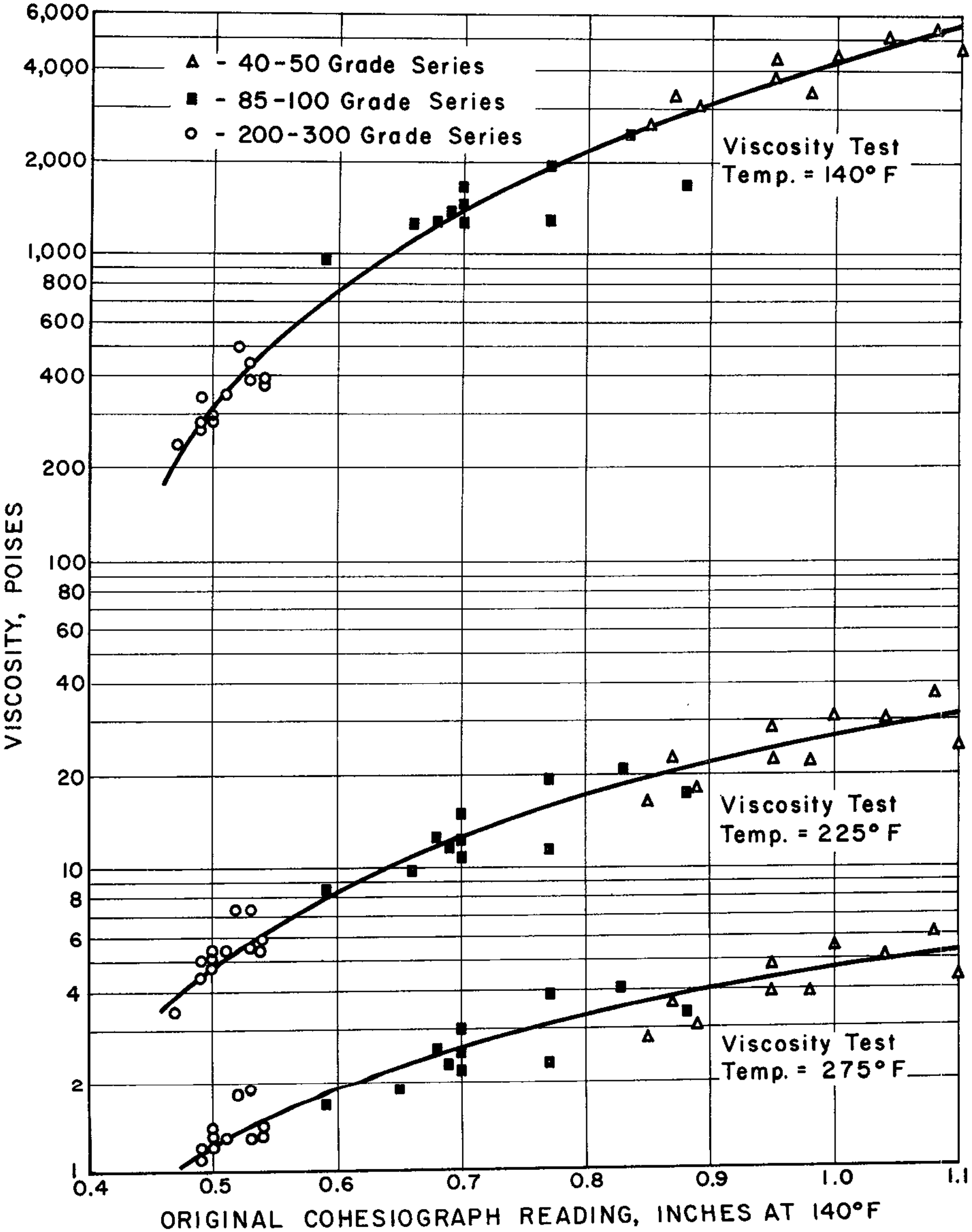


FIGURE 7

LABORATORY MIXER CALIBRATION
29 PROJECTS

Mixing Time = 3 Min

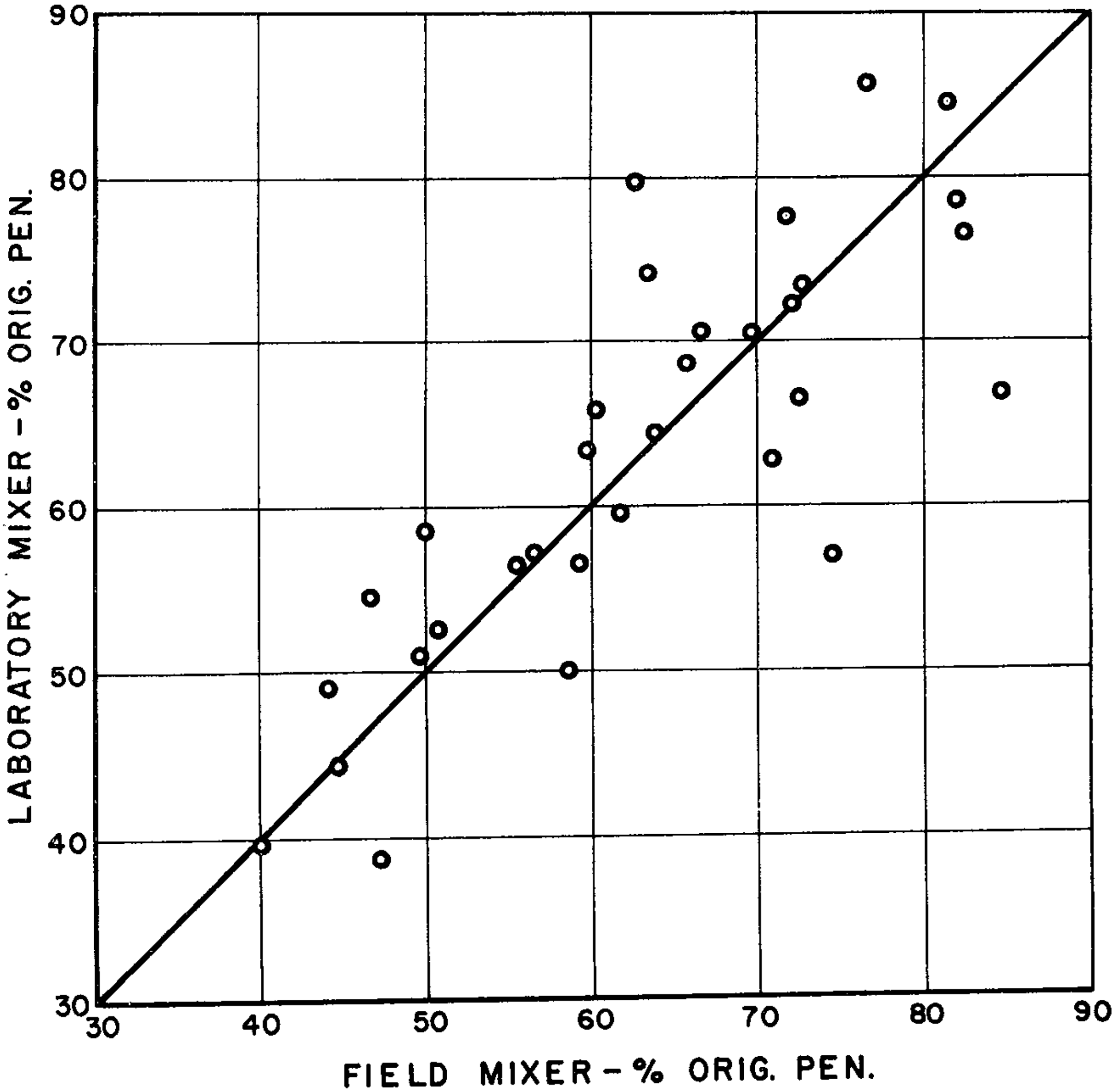


FIGURE 8

TYPICAL WEATHERING MACHINE CALIBRATION CURVE
CONT. 52-4TC10

120-150 Paving Asphalt

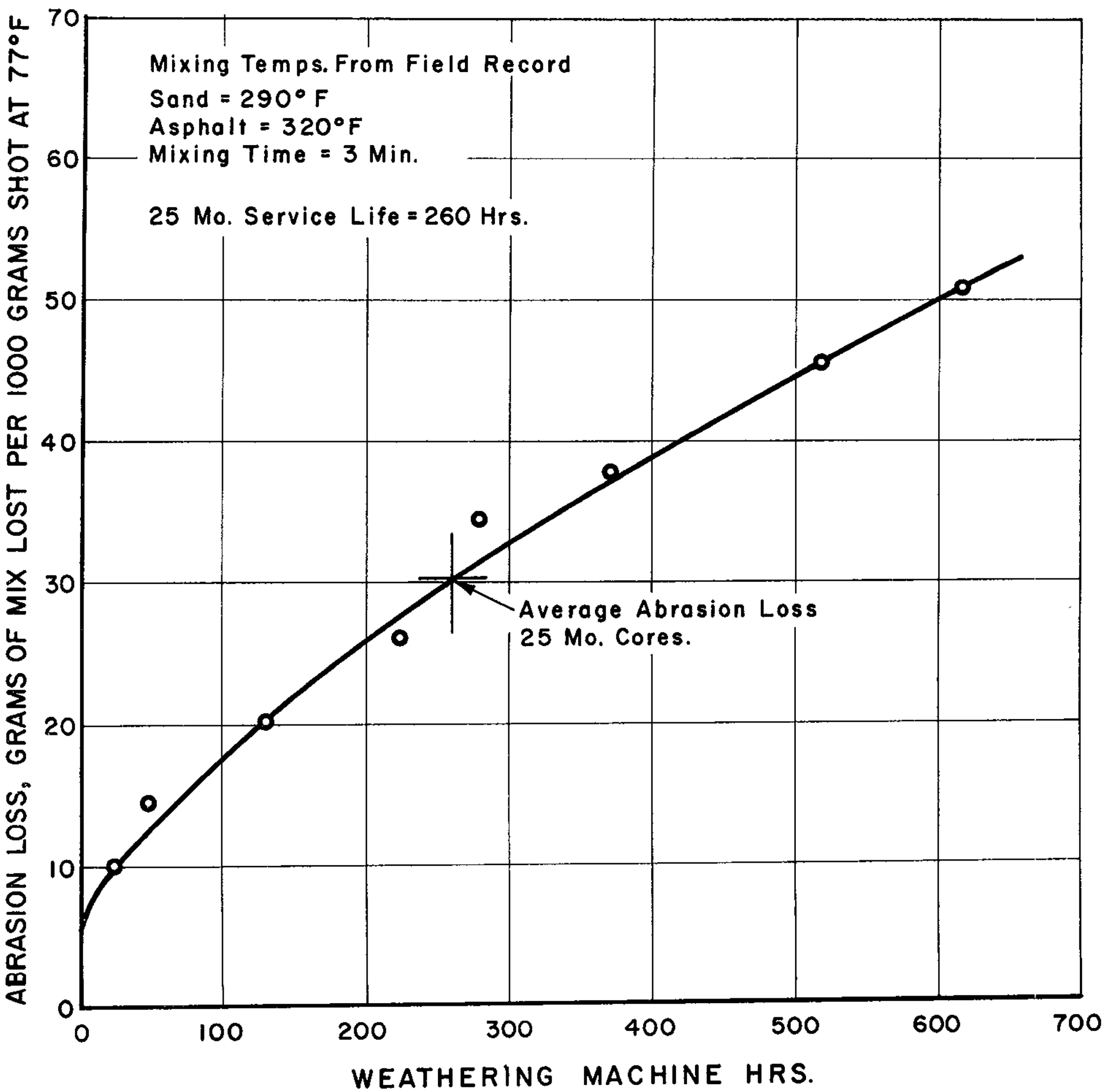


FIGURE 9

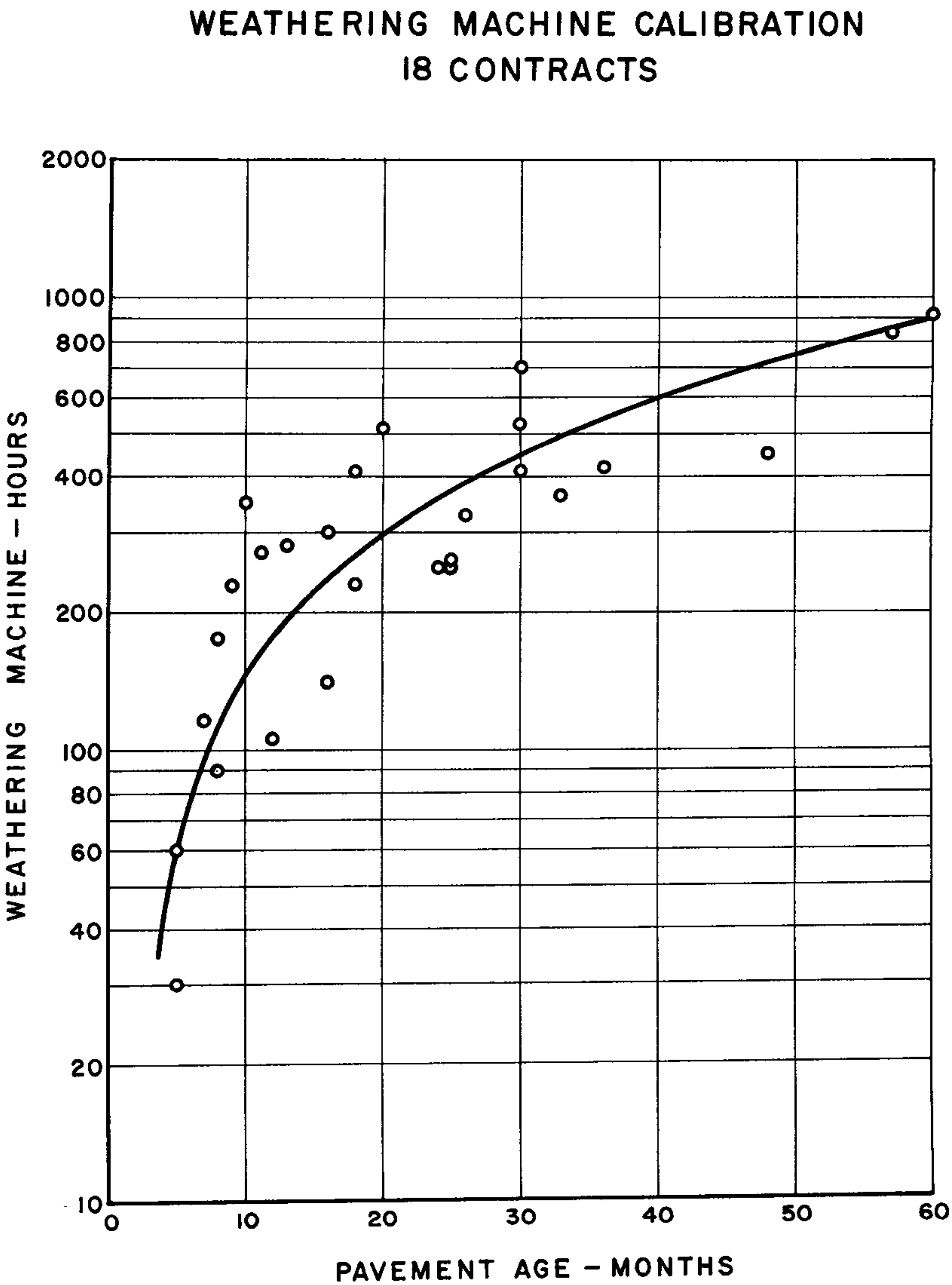

WEATHERING MACHINE CALIBRATION
18 CONTRACTS
WEATHERING MACHINE — HOURS
2000
1000
800
600
400
200
100
80
60
40
20
10
0
10
20
30
40
50
60
PAVEMENT AGE — MONTHS

FIGURE 10

WEATHERING MACHINE CALIBRATION
ZACA-WIGMORE PROJECT

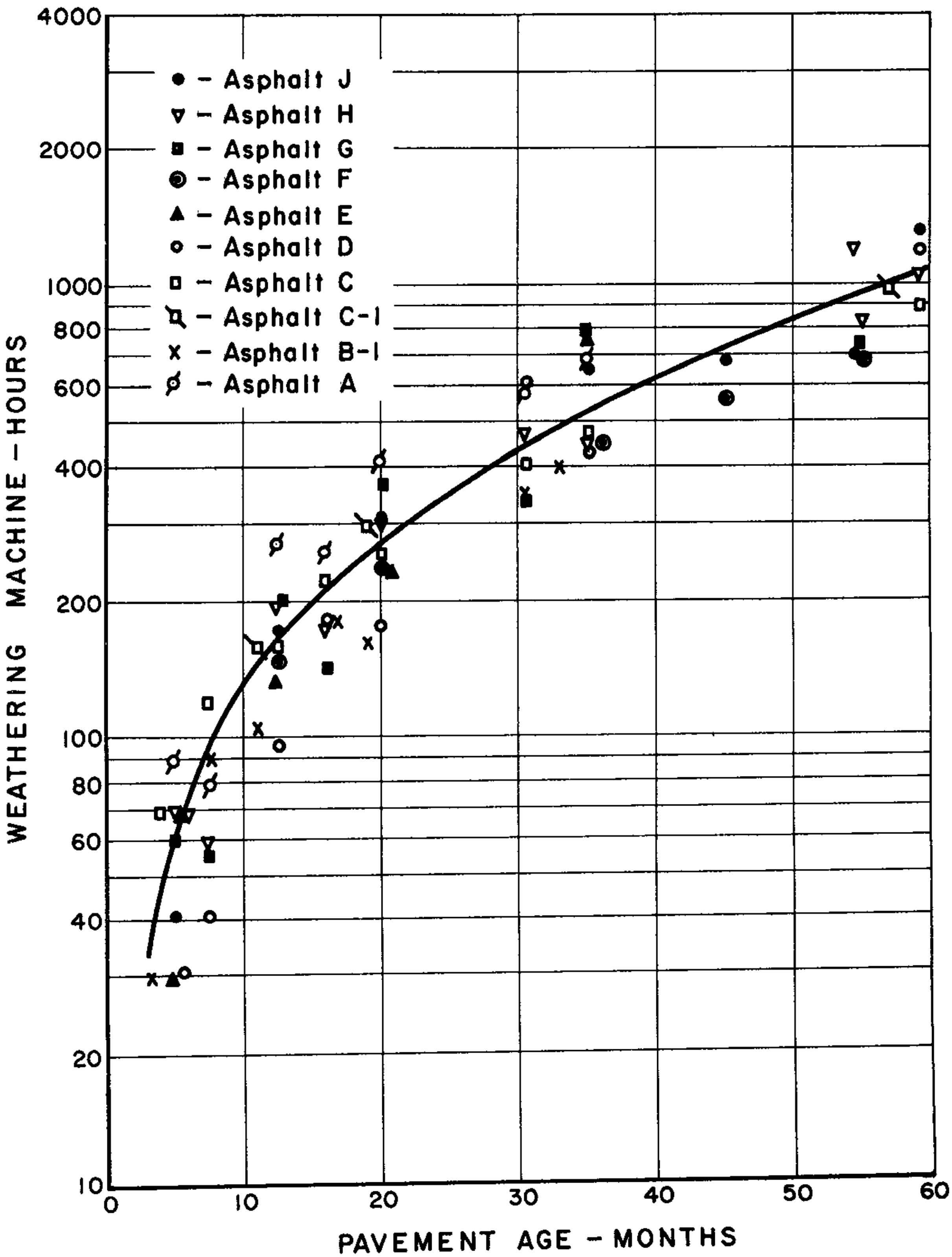


FIGURE 11

WEATHERING MACHINE CALIBRATION CURVES

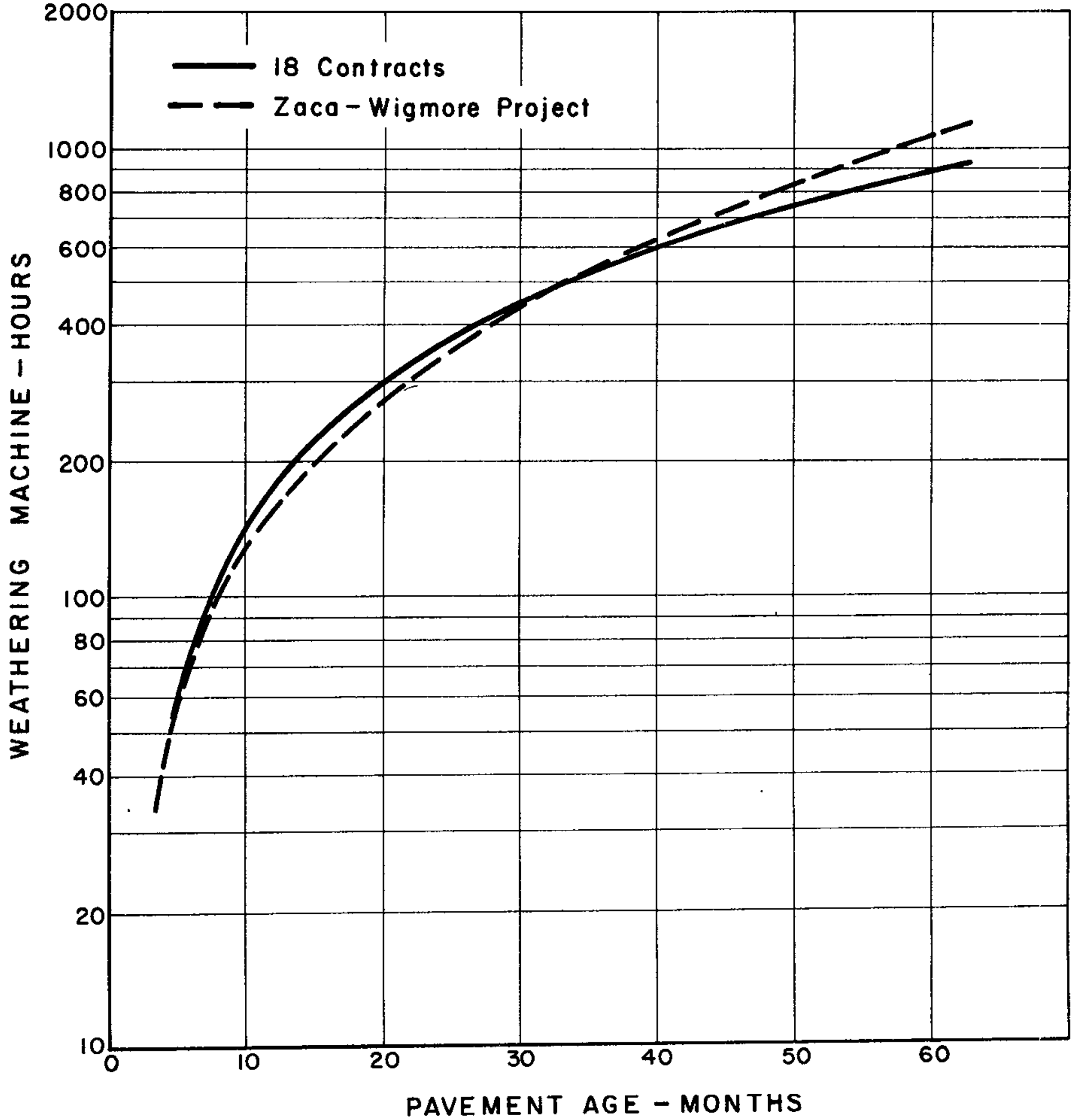


FIGURE 12

ABRASION TEST RESULTS FOR VARIOUS 85-100 GRADE PAVING ASPHALTS

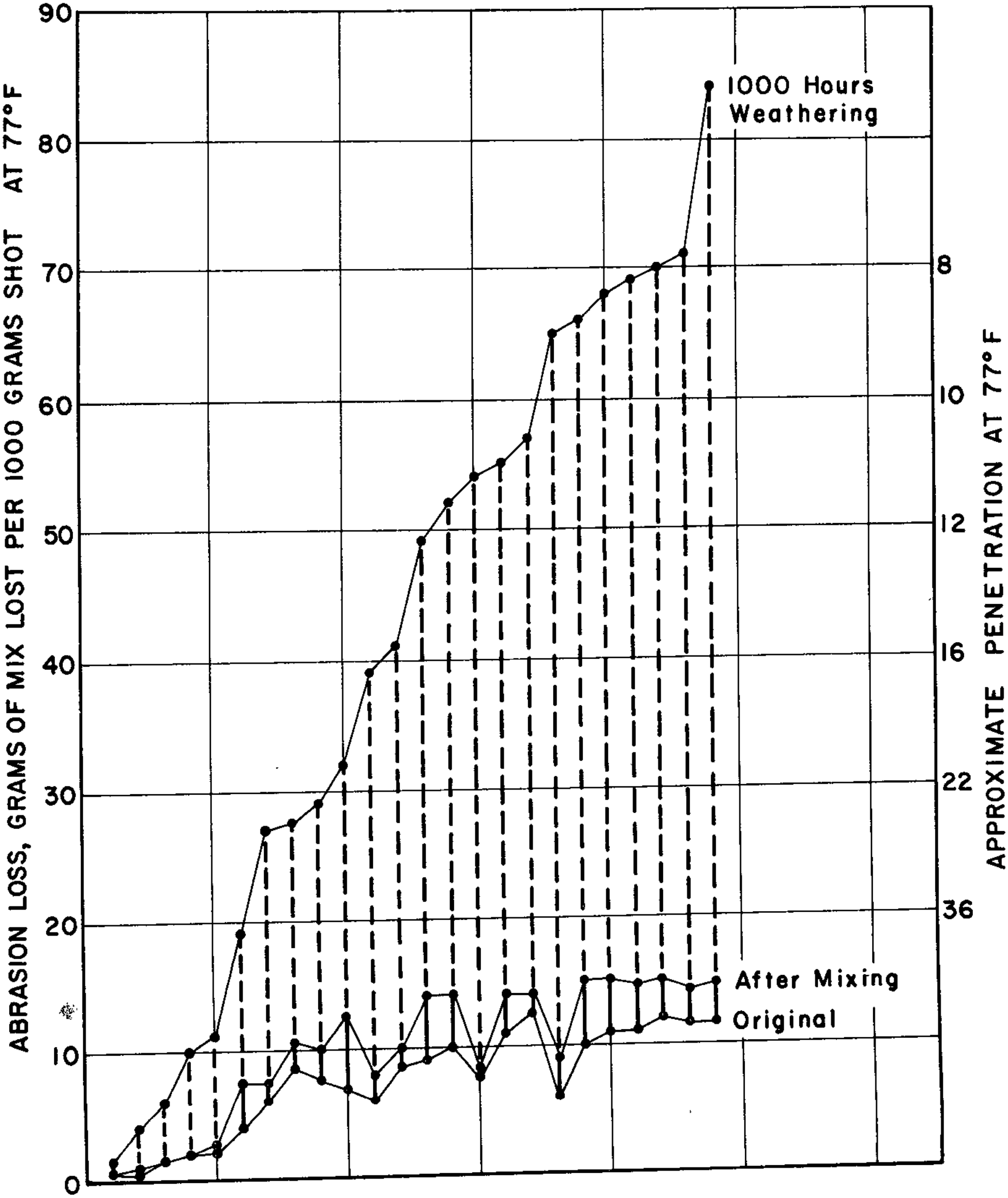


FIGURE 13

CHANGE IN VISCOSITY DURING WEATHERING OF VARIOUS 85-100 GRADE PAVING ASPHALTS

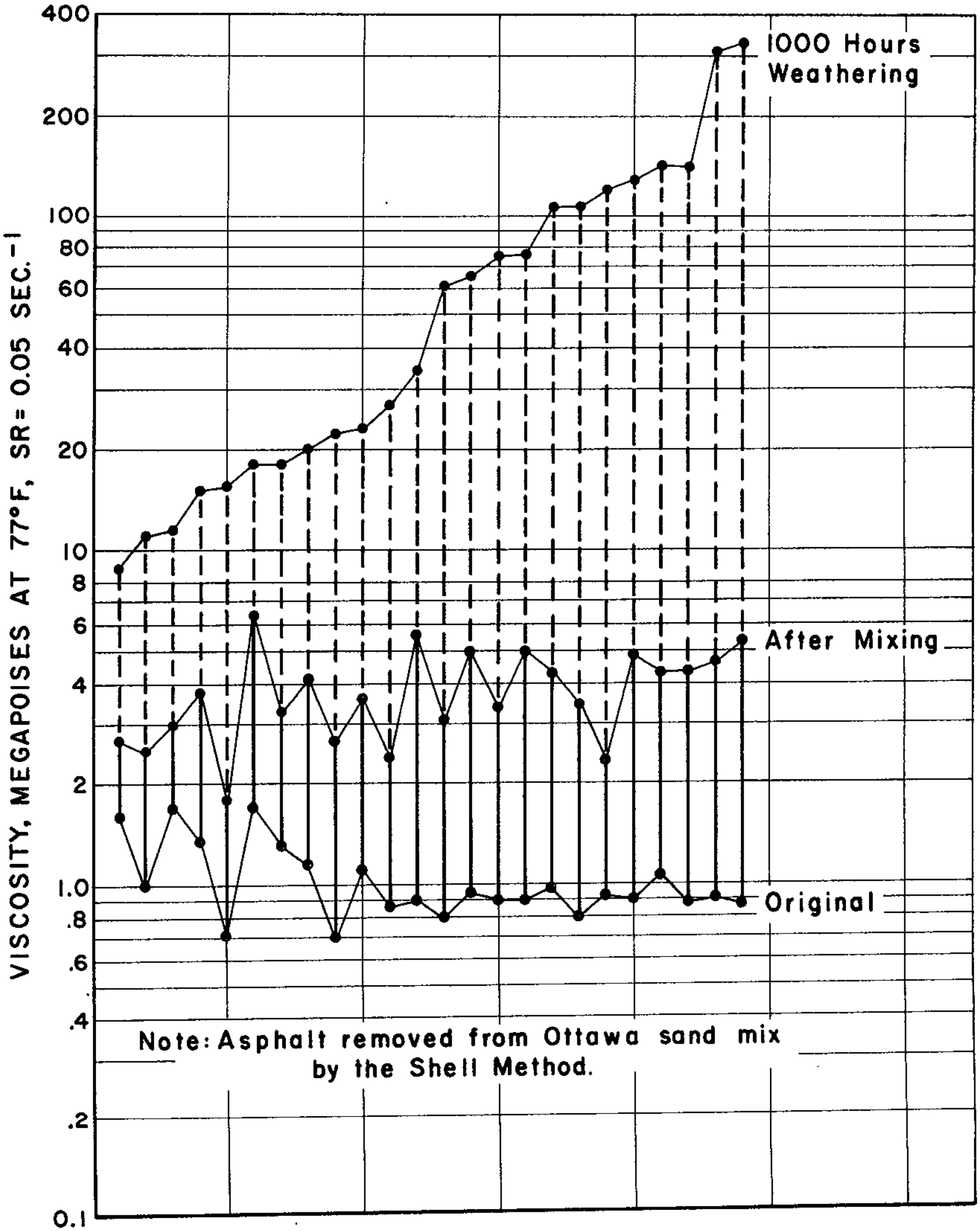


FIGURE 14

CHANGE IN SHEAR SUSCEPTIBILITY DURING WEATHERING FOR VARIOUS 85-100 GRADE PAVING ASPHALTS

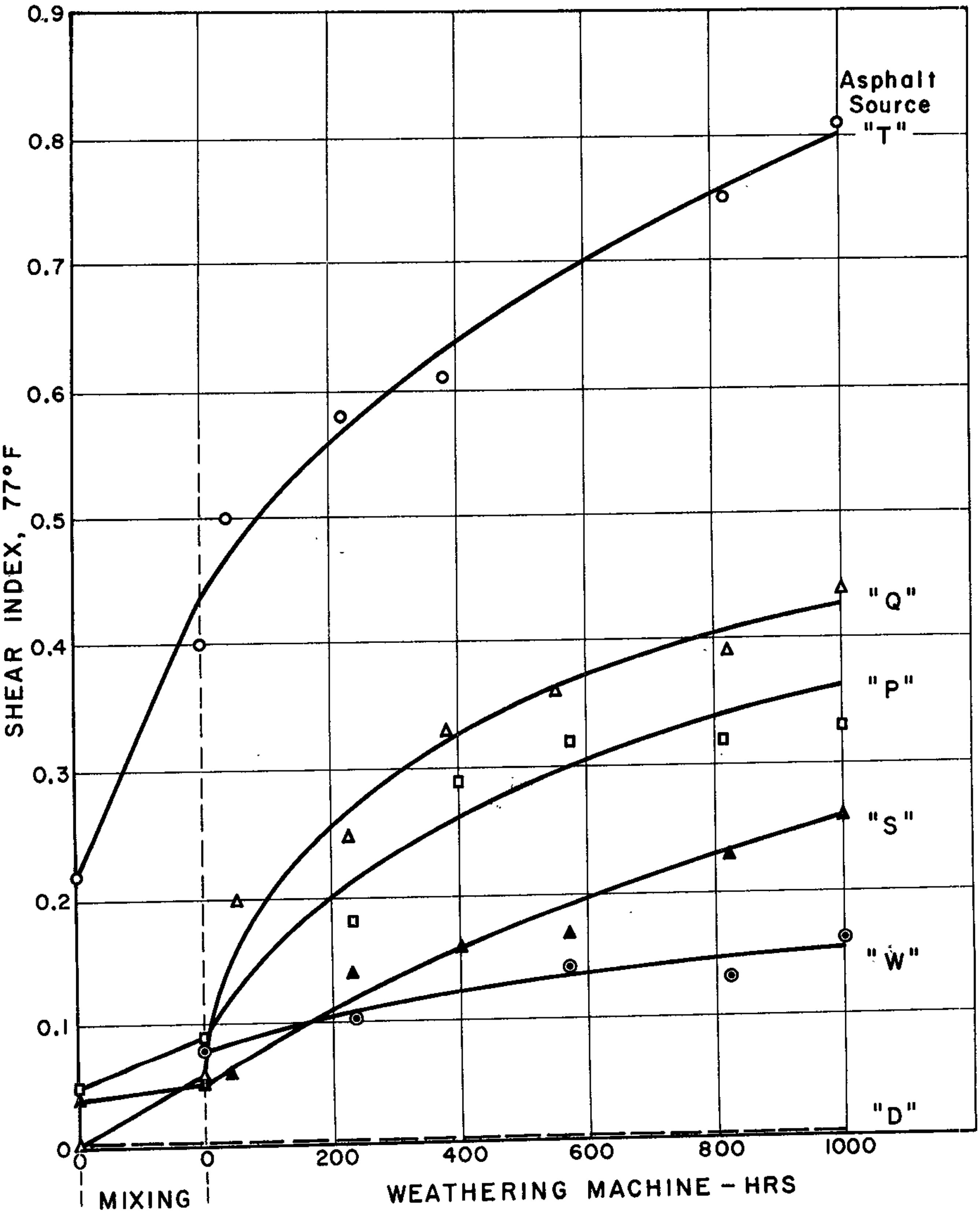


FIGURE 15

ROLLING THIN FILM TEST CALIBRATION
15 PROJECTS

Test Period = 73 Min.

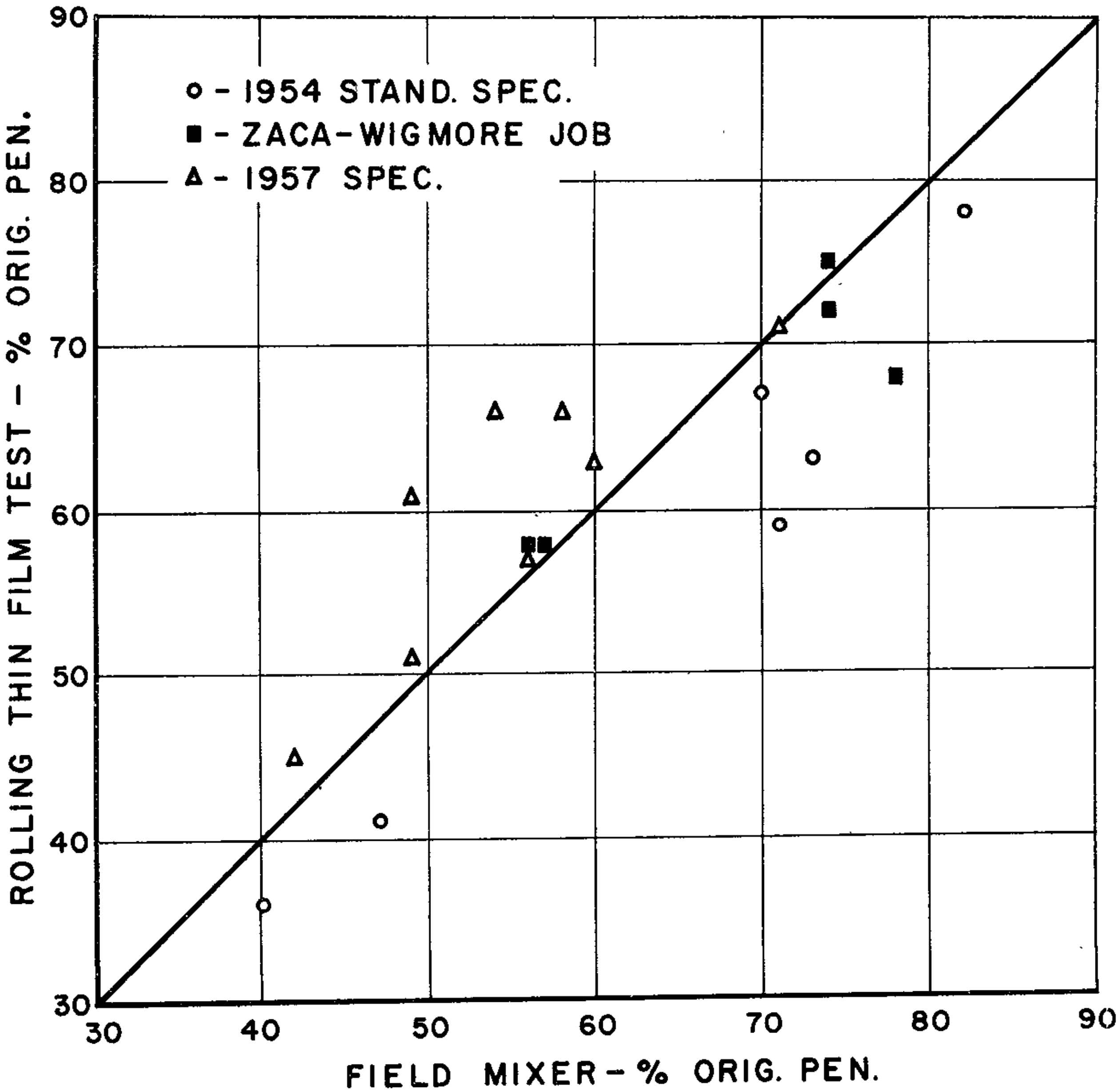


FIGURE 16

CORRELATION OF OTTAWA SAND MIXER AND ROLLING THIN FILM TEST

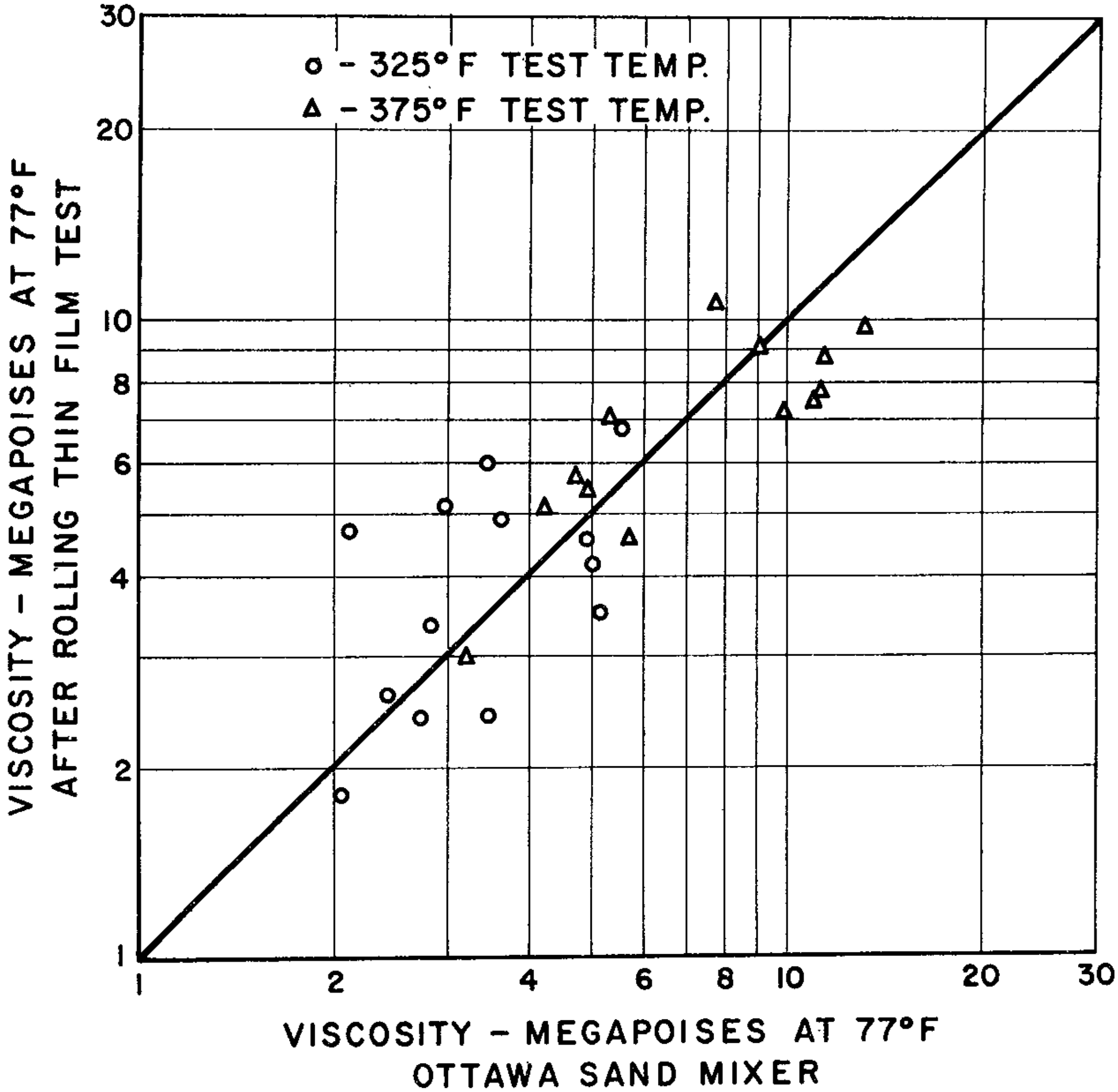


FIGURE 17

ROLLING THIN FILM TEST, LEWIS THIN FILM TEST RELATION

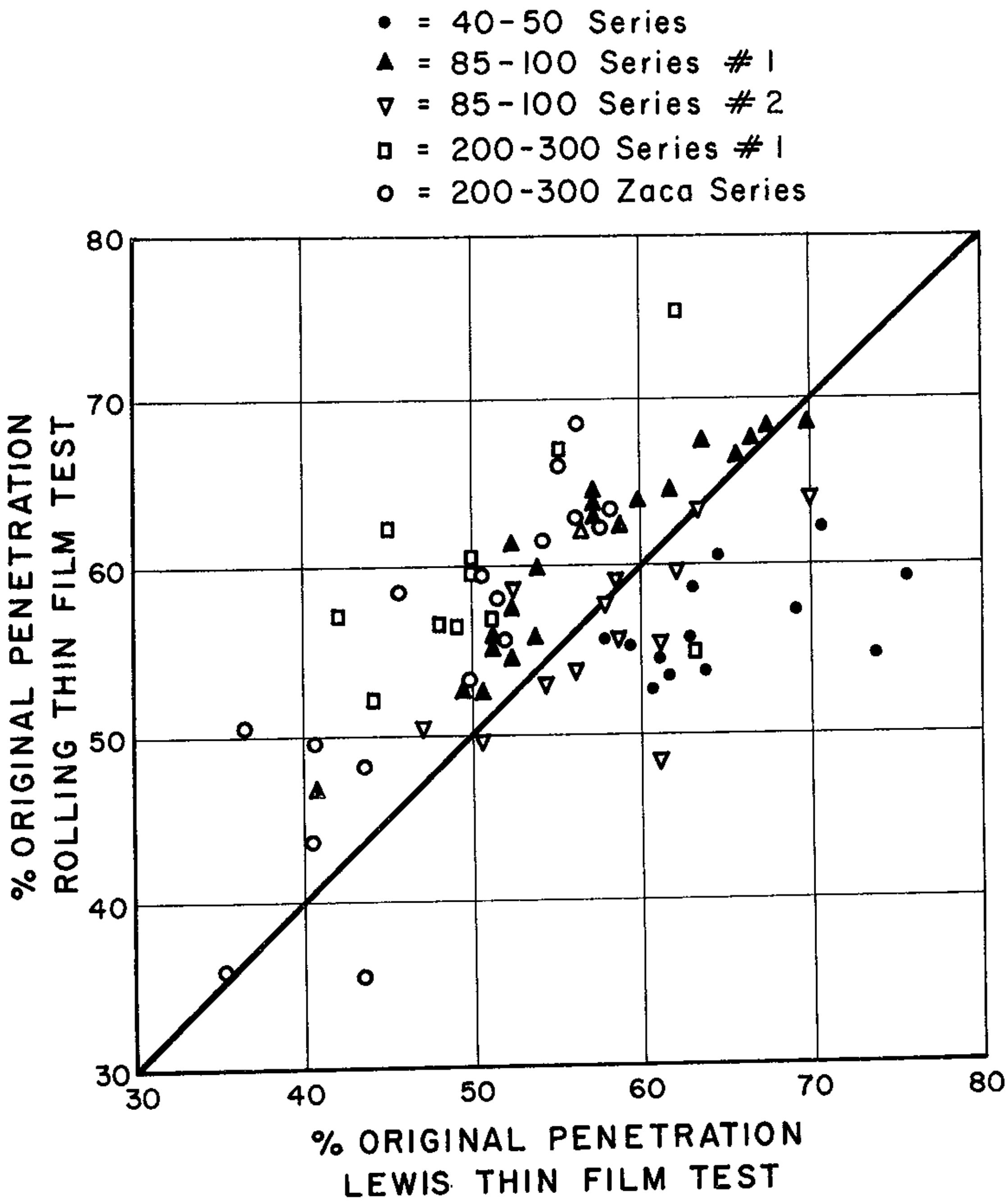


FIGURE 18

CORRELATION OF VISCOSITY AFTER 1000 HOURS IN THE WEATHERING UNIT WITH VISCOSITY AFTER 24 HOURS AT 210°F IN 20 MICRON FILMS

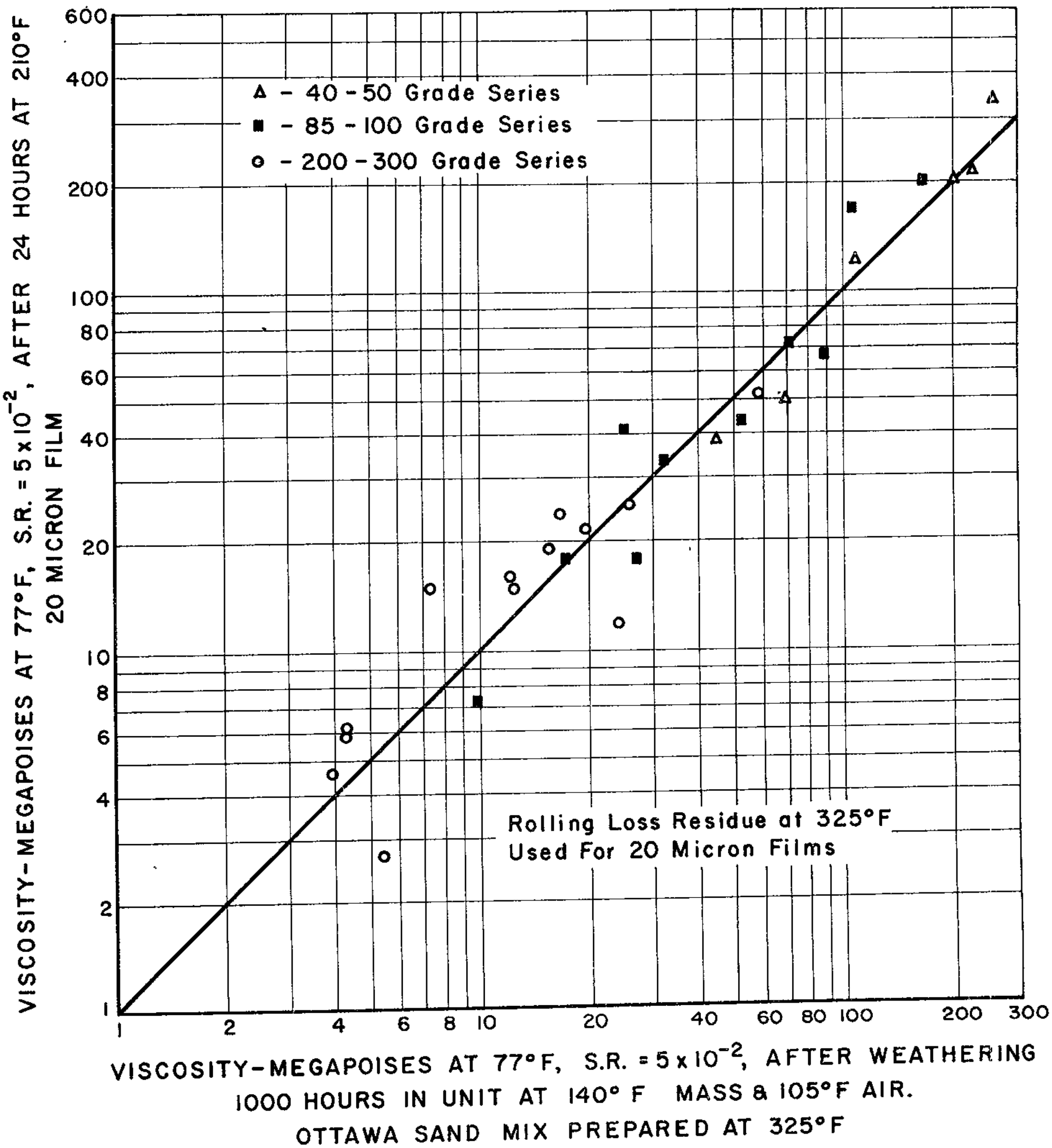


FIGURE 19

CHANGE IN SHEAR SUSCEPTIBILITY DURING WEATHERING - DOYLE 85-100 TEST SERIES

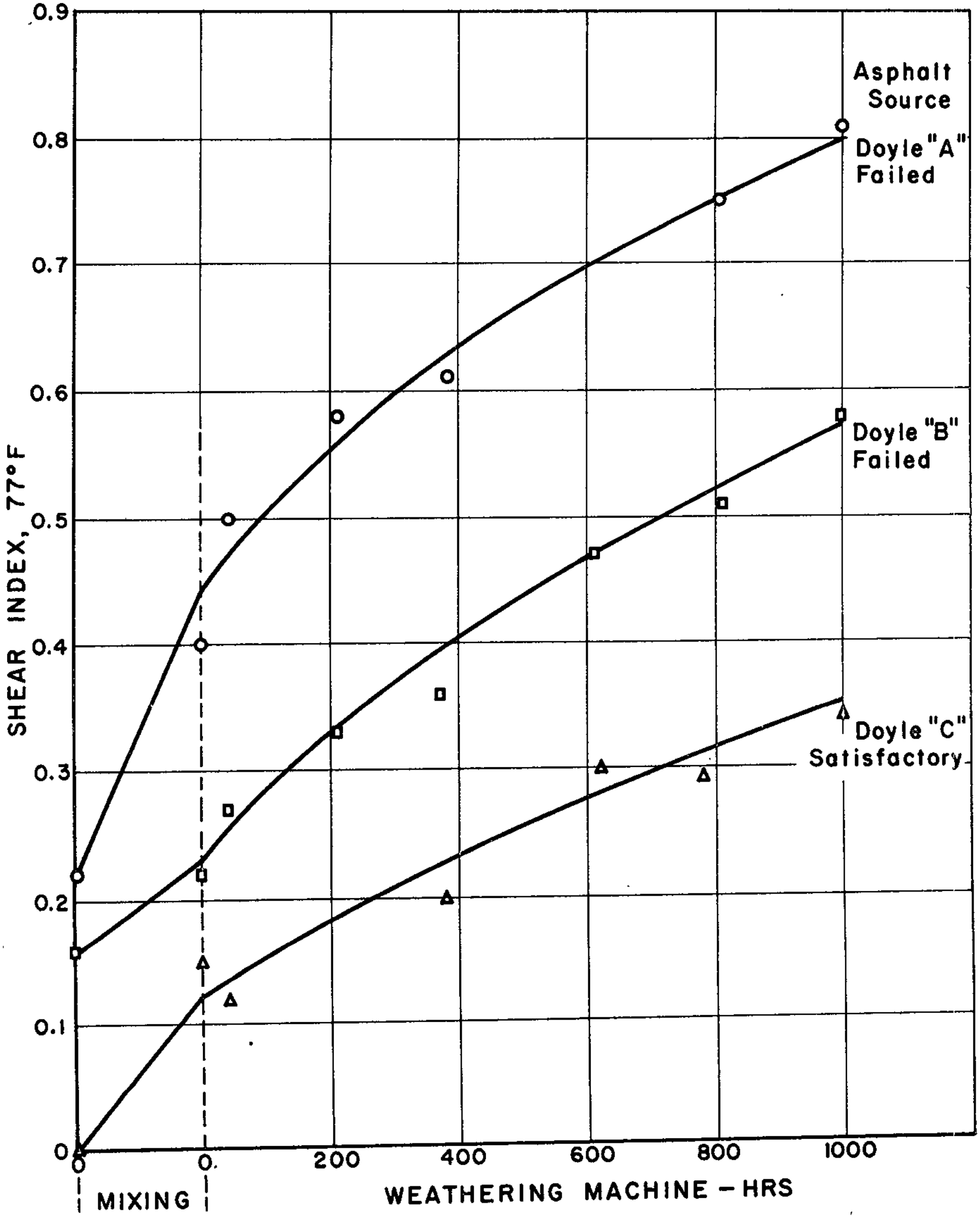
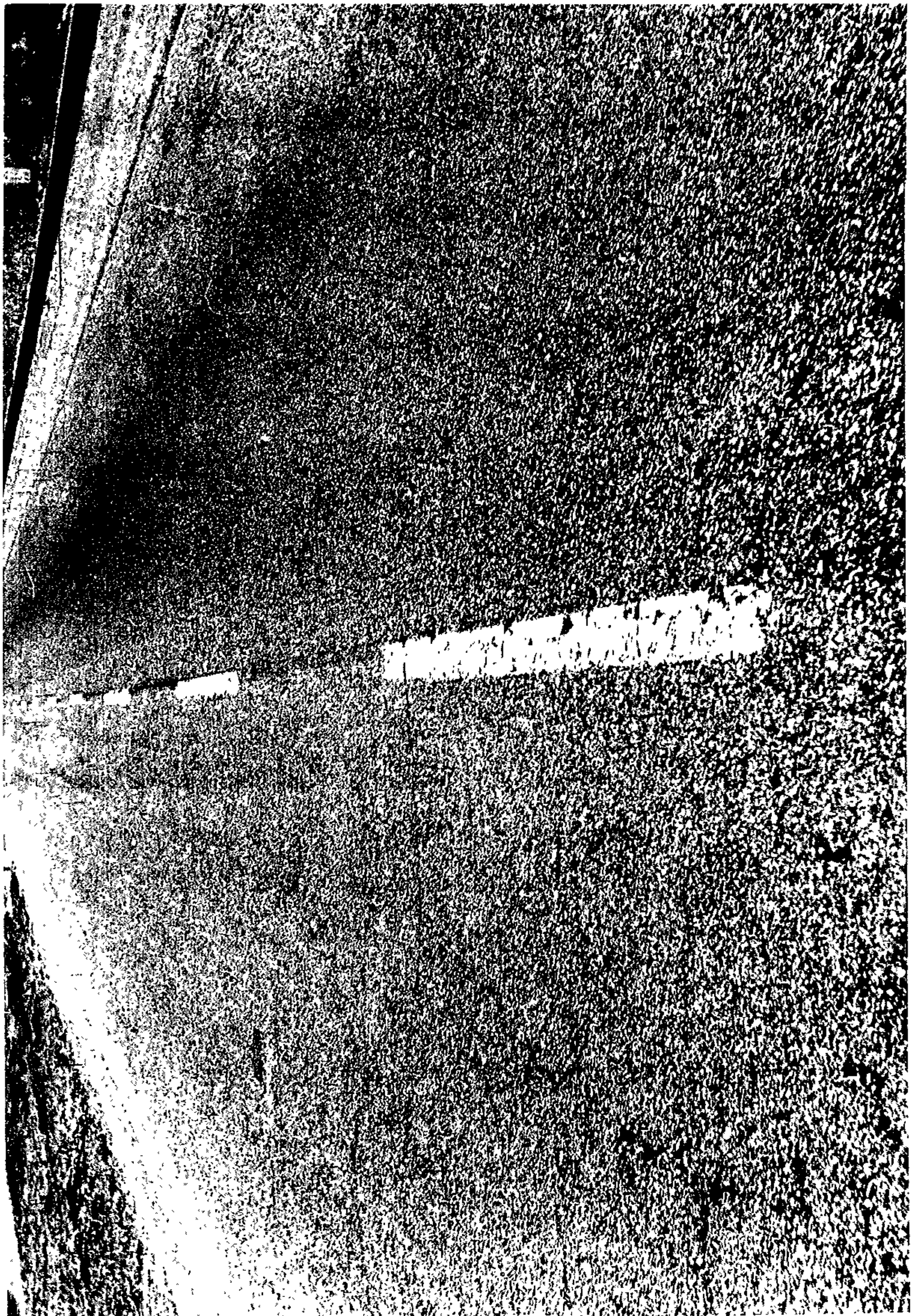


FIGURE 20



Pavement Condition of Asphalt I Test Section After 82 Months of Service Life; Zaca - Wigmore Project

FIGURE 21

SHEAR SUSCEPTIBILITY CURVES AFTER 1000 HOURS WEATHERING

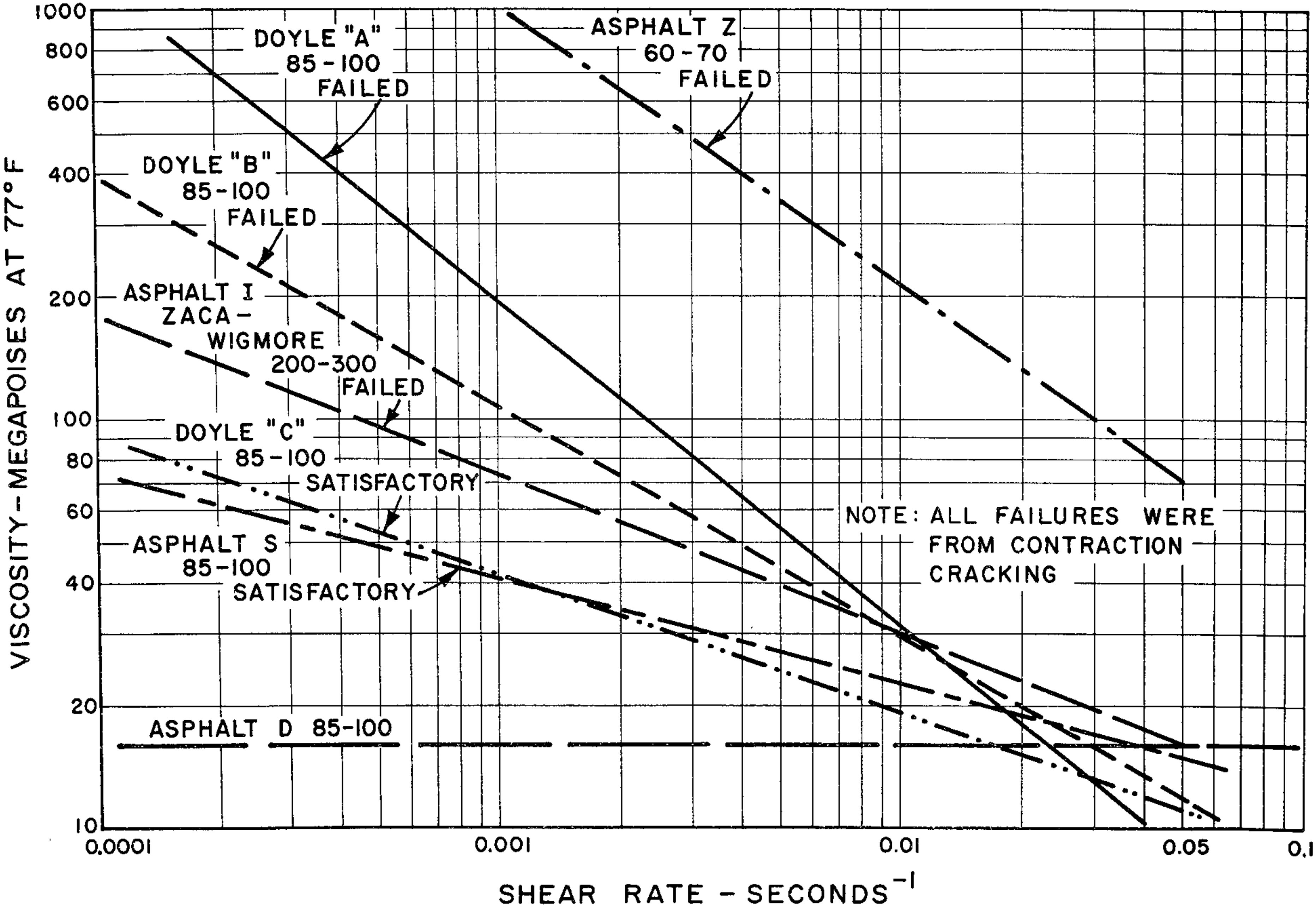


FIGURE 22

EFFECT OF TIME ON STAIN NUMBER
FOR VARIOUS 85-100 GRADE PAVING ASPHALTS

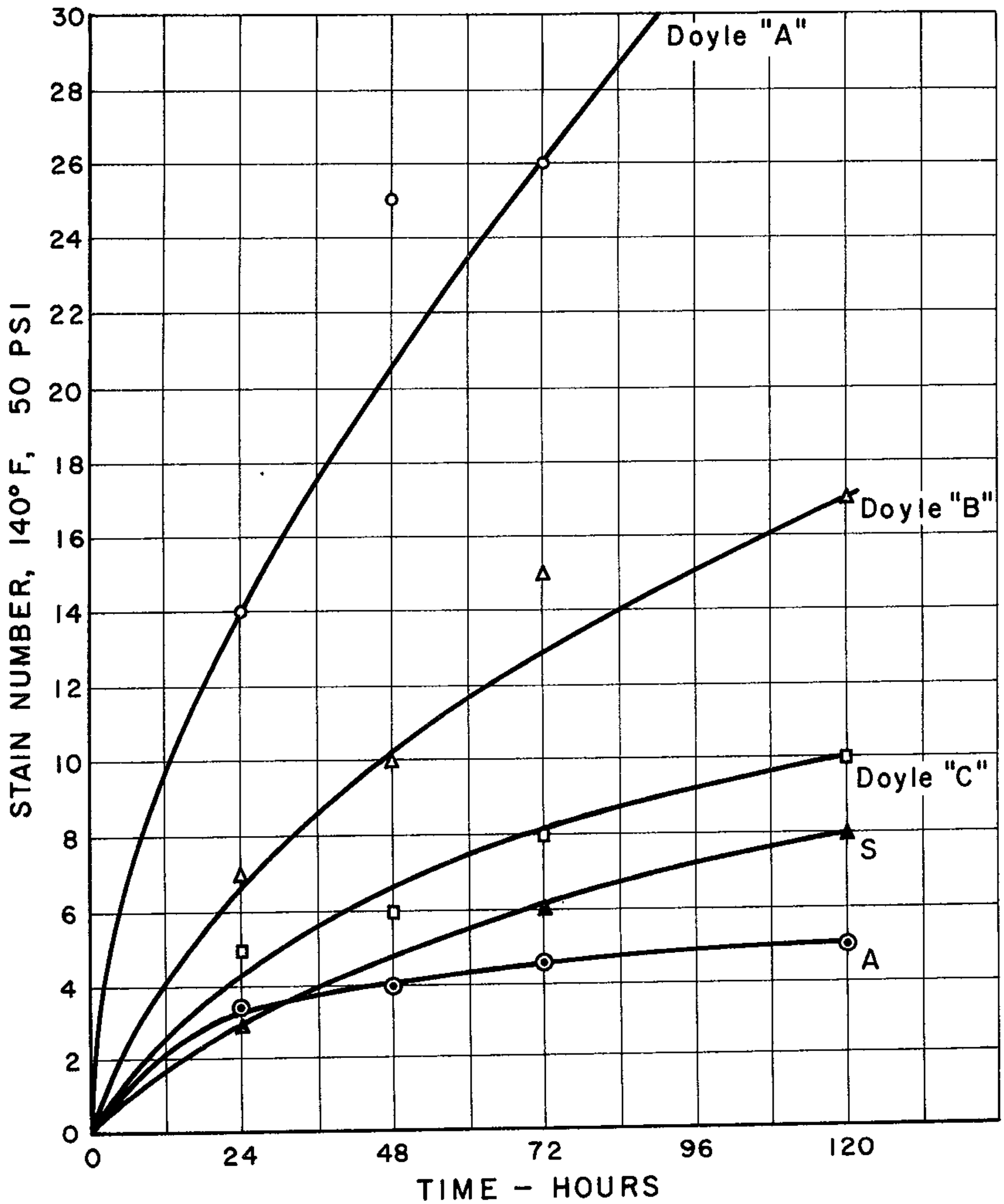
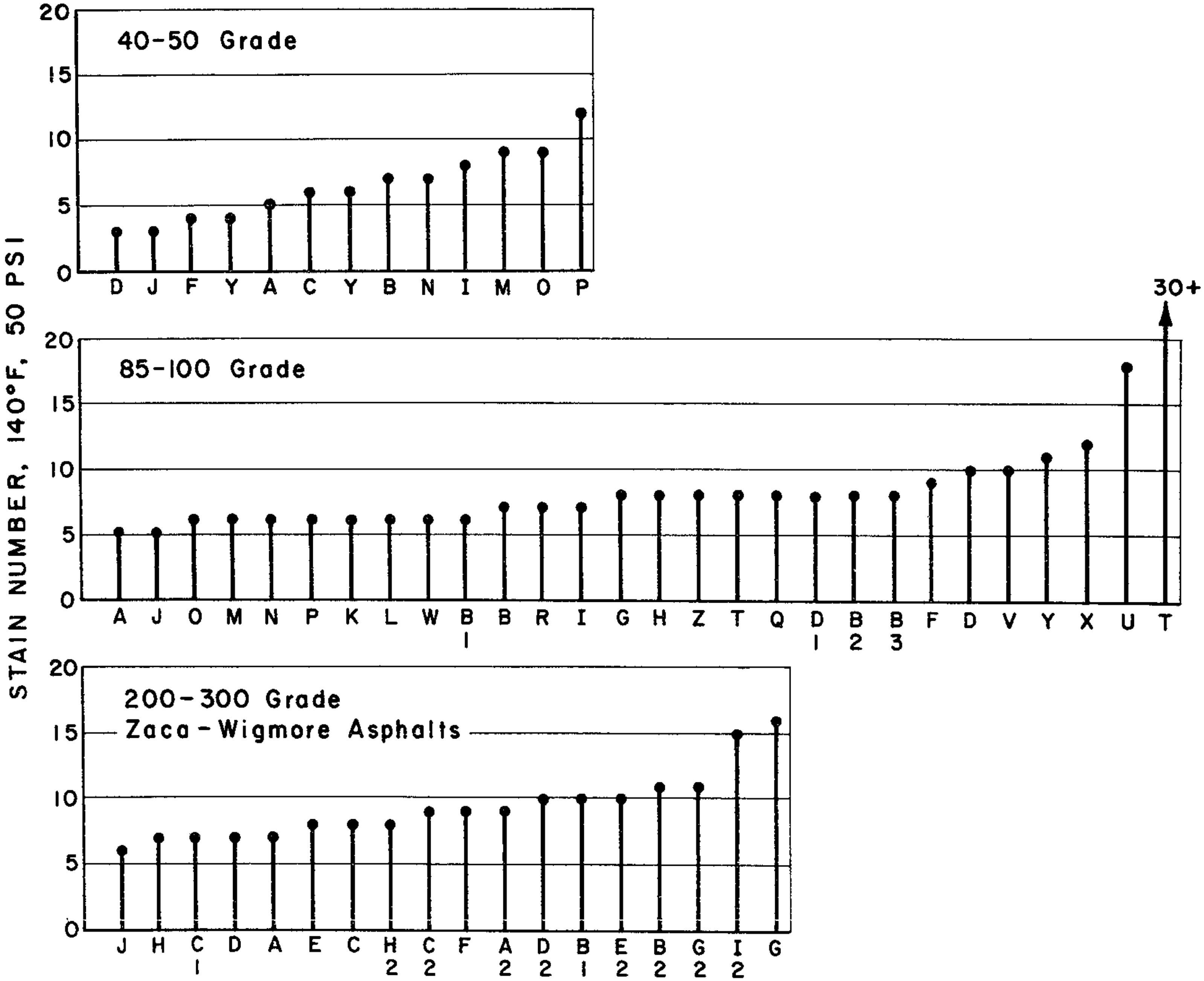


FIGURE 23

STAIN NUMBER FOR VARIOUS GRADES OF PAVING ASPHALT



APPENDIX

A. Tentative Method of Test for Determining the "Setting" Characteristics of Paving Grade Asphalts.

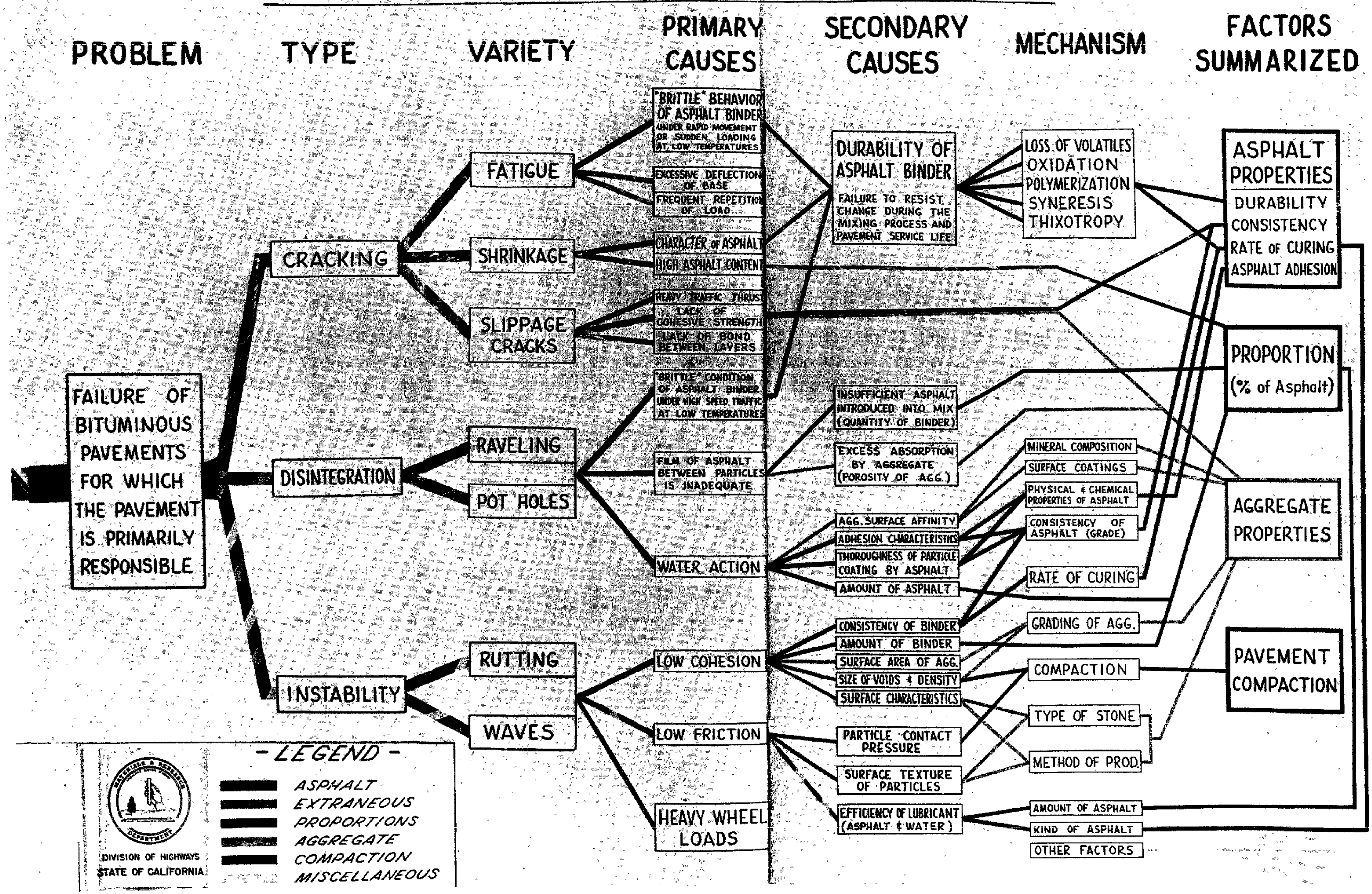
B. Preparation of 2% Asphalt Ottawa Sand Mix by the Solvent Method.

C. Tentative Method of Test for Simulating the Effect of Field Mixing on Paving Grade Asphalts, (Rolling Thin Film Test).

D. Tentative Method of Test for Determining the Effect of Heat and Air on Paving Grade Asphalts in Thin Films.

E. Tentative Method of Test for Determination of Viscosity of Bituminous Materials by Means of the Microviscometer.

F. Tentative Method of Test for Micro-Ductility of Bituminous Materials.



PROBLEM
TYPE
VARIETY
PRIMARY CAUSES
SECONDARY CAUSES
MECHANISM
FACTORS SUMMARIZED
FAILURE OF BITUMINOUS PAVEMENTS FOR WHICH THE PAVEMENT IS PRIMARILY RESPONSIBLE
CRACKING
DISINTEGRATION
INSTABILITY
FATIGUE
SHRINKAGE
SLIPPAGE CRACKS
RAVELING
POT HOLES
RUTTING
WAVES
"BRITTLE" BEHAVIOR OF ASPHALT BINDER UNDER RAPID MOVEMENT OR SUDDEN LOADING AT LOW TEMPERATURES
EXCESSIVE DEFLECTION OF BASE
FREQUENT REPETITION OF LOAD
CHARACTER OF ASPHALT
HIGH ASPHALT CONTENT
HEAVY TRAFFIC THRUST
LACK OF COHESIVE STRENGTH
LACK OF BOND BETWEEN LAYERS
"BRITTLE" CONDITION OF ASPHALT BINDER UNDER HIGH SPEED TRAFFIC AT LOW TEMPERATURES
FILM OF ASPHALT BETWEEN PARTICLES IS INADEQUATE
WATER ACTION
LOW COHESION
LOW FRICTION
HEAVY WHEEL LOADS
DURABILITY OF ASPHALT BINDER
FAILURE TO RESIST CHANGE DURING THE MIXING PROCESS AND PAVEMENT SERVICE LIFE
INSUFFICIENT ASPHALT INTRODUCED INTO MIX (QUANTITY OF BINDER)
EXCESS ABSORPTION BY AGGREGATE (POROSITY OF AGG.)
AGG. SURFACE AFFINITY
ADHESION CHARACTERISTICS
THOROUGHNESS OF PARTICLE COATING BY ASPHALT
AMOUNT OF ASPHALT
CONSISTENCY OF BINDER
AMOUNT OF BINDER
SURFACE AREA OF AGG.
SIZE OF VOIDS & DENSITY
SURFACE CHARACTERISTICS
PARTICLE CONTACT PRESSURE
SURFACE TEXTURE OF PARTICLES
EFFICIENCY OF LUBRICANT (ASPHALT & WATER)
LOSS OF VOLATILES
OXIDATION
POLYMERIZATION
SYNERESIS
THIXOTROPY
MINERAL COMPOSITION
SURFACE COATINGS
PHYSICAL & CHEMICAL PROPERTIES OF ASPHALT
CONSISTENCY OF ASPHALT (GRADE)
RATE OF CURING
GRADING OF AGG.
COMPACTION
TYPE OF STONE
METHOD OF PROD.
AMOUNT OF ASPHALT
KIND OF ASPHALT
OTHER FACTORS
ASPHALT PROPERTIES
DURABILITY
CONSISTENCY
RATE OF CURING
ASPHALT ADHESION
PROPORTION
(% of Asphalt)
AGGREGATE PROPERTIES
PAVEMENT COMPACTION
- LEGEND -
ASPHALT
EXTRANEOUS
PROPORTIONS
AGGREGATE
COMPACTION
MISCELLANEOUS
DIVISION OF HIGHWAYS
STATE OF CALIFORNIA

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

TENTATIVE METHOD OF TEST FOR DETERMINING THE "SETTING" CHARACTERISTICS OF PAVING GRADE ASPHALTS

Scope

This method of test provides a means for the determination of the "setting" rates of paving grade asphalts.

Procedure

A. Apparatus

1. Oven - adequate size; well ventilated by convection currents of air; capable of maintaining a temperature of 140±2F.

2. Mixer - designed for laboratory usage. Detailed plans are available from the Materials and Research Department.

3. Cohesiograph - a unit for determining the "setting" rates of asphalts. Detailed plans of the Cohesiograph and its accessories are available from the Materials and Research Department.

4. Hot Plates

   a. Small type (6"x6") continuously adjustable, to maintain a temperature above 325F.

   b. Large type, continuously adjustable, to maintain a temperature of 325F by thermostat.

5. Balances

   a. Small, 200 gram capacity, .01 gm. sensitivity.

   b. Large, 1000 gram capacity, 1 gm. sensitivity.

6. Assorted pans

   a. Four (4) small; diameter, 5-1/2"; depth, 1-1/2".

   b. Two (2) special 3 Oz. tins with pouring spout and tab handle.

7. Miscellaneous Equipment

   a. Timer, sweep second hand.
   b. Timer, 60 minute with bell.
   c. Medium cement trowel 4".
   d. Small cement trowel 3".
   e. Thermometer, ASTM #16F, high softening point.

B. Material

1. Ottawa Sand #20-30, A.S.T.M. C-190

2. Paper liners 1000H approximately 1-7/8" x 11-7/8".

3. Adding machine paper, 3" wide.

C. Preparation of Sample

1. Preparation of Sample for Mixing.

Weigh two (2) 1000 gram samples of 20-30 mesh Ottawa sand. Pour one of the samples of sand into each of the two mixer bowls. Heat the sand continuously in the mixer until it attains a temperature of 325F. Near the end of the preheating operation, switch on the mixer to stir the sand and insure a uniform sand temperature.

While the sand is being heated to the proper temperature, weigh duplicate portions of the hot asphalt sample into the special 3-oz. tins. Measure enough asphalt so that there will be a 2.0±.05% asphalt mixture after mixing with the 1000 gram portion of sand. The hold-up in the tin varies with each asphalt and a trial pouring to determine hold-up may be necessary. For example: Assume a hold-up in the tin of 1.50 gms., then; 20 gms. (for mix) plus 2 gms. (for hold-up in mixer) = 20.00+2.00+1.50 or 23.50 gms. (in 3-oz. tin).

2. Mixing Procedure

   a. Adjust the larger hot plate to the temperature of 325F and the smaller one to a slightly higher temperature.

   b. Place one of the tins of previously weighed asphalt upon the small hot plate. When the asphalt becomes almost entirely melted, stir the asphalt with the thermometer while holding the tin with tongs.

   c. Carefully watch the temperature until it reaches 325F, then place the sample on the larger hot plate, set at 325F, and wipe off the thermometer on the lip of the tin. Immediately pour all of the asphalt into the mixer (with mixer in operation) in exactly 10 seconds time (Figure I).

d. While the asphalt is being mixed with the sand for a three minute period, reweigh the asphalt container to determine the amount of retained material.

e. After mixing for three minutes, deposit the asphalt-sand mixture into a large pan. Discard any mixture that clings to the sides of the mixing bowl.

f. Level the mixture uniformly and place a cover on the pan to retain the heat.

g. Repeat the above heating and mixing procedure for the duplicate asphalt sample, remove cover from pan, deposit with previously mixed sample; then combine the two mixes with 60 strokes of the medium trowel.

3. Preparation of Sample for Testing

a. Weigh four (4) 220 gram portions of the asphalt-Ottawa sand mixture into the 5-1/2 inch pans, place these pans in an oven set at 140F and leave in oven one hour.

b. After the sample has reheated for one hour, remove from oven.

c. Position the brass mold in the mold-holder and wipe the mold clean using a rag containing a small amount of kerosene. Line the mold with a paper strip so that the mixture will not stick to the mold.

d. Knead and compact the entire 220 gram portion into the mold by moving the face of the small trowel back and forth over the surface of the mixture. Occasionally wipe the trowel surface with a rag containing a slight amount of kerosene to prevent sticking.

e. Proceed in this manner until the sample is smooth and flush with the edges of the mold, (Figure 2).

f. Mold four (4) of these samples, and test two (2) of these in the Cohesiograph, immediately after molding. Place the other two (2) samples in an oven set at 140F for testing 24 hours after molding.

D. Testing Procedure

1. Remove the paper liner by carefully inverting the sample and mold on a board, (Figure 3). Wipe the mold with the kerosene rag and place mold over top of sample and invert.

2. Place the sample in the Cohesiograph and lower the heavy heating plate with the dial-type temperature gauges, onto the sample. Position the sample with the mold follower in place, (Figure 4), and allow sample to return to 140F. This period of time shall be at least 10 minutes.

   Note: Cohesiograph must be turned on at least two hours in advance of testing time.

3. After the sample has attained a temperature of 140F, position the pen on the movable portion of the Cohesiograph. Test its operation by making trial marks on the paper.

4. Turn the motor on and engage the clutch so that the sample will be extruded and broken off in chunks, thus, striking the arm of the marking pen and plotting breaks on the paper.

5. Determine the average length of break by measuring the total length of breaks and dividing this distance by the total number of breaks. Abnormal breaks are disregarded in this determination.

6. The final determination is the average length of break of the duplicate samples. For example: Let us assume a total length of break of 8.250" and a total number of 10 breaks. The average length of break would then be 0.825". Let us again assume an average length of break of 0.835" for the duplicate sample. The final determination or the average of the two samples would then be 0.830".

REFERENCE
A California Method
End of Text on Calif. 350-A

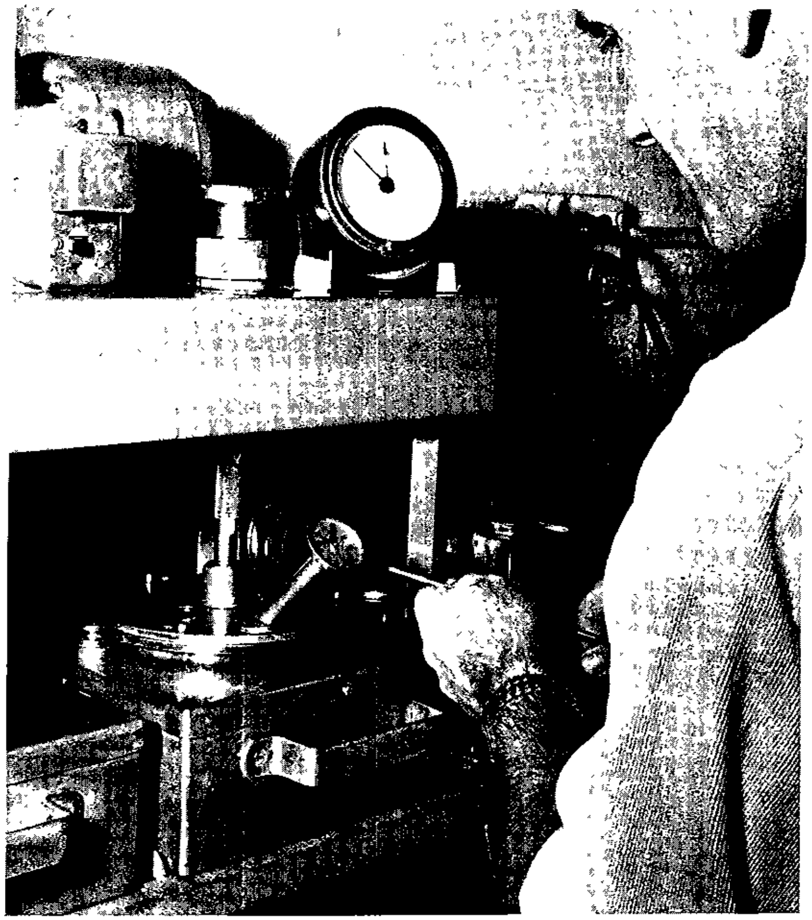


Figure I
Pouring Asphalt Into Mixer

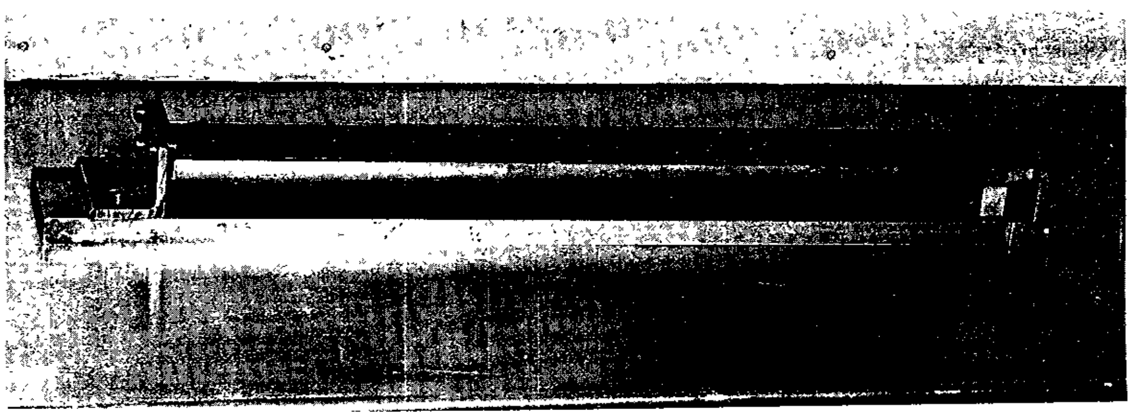


Figure II
Asphalt Sand Mixture After Molding

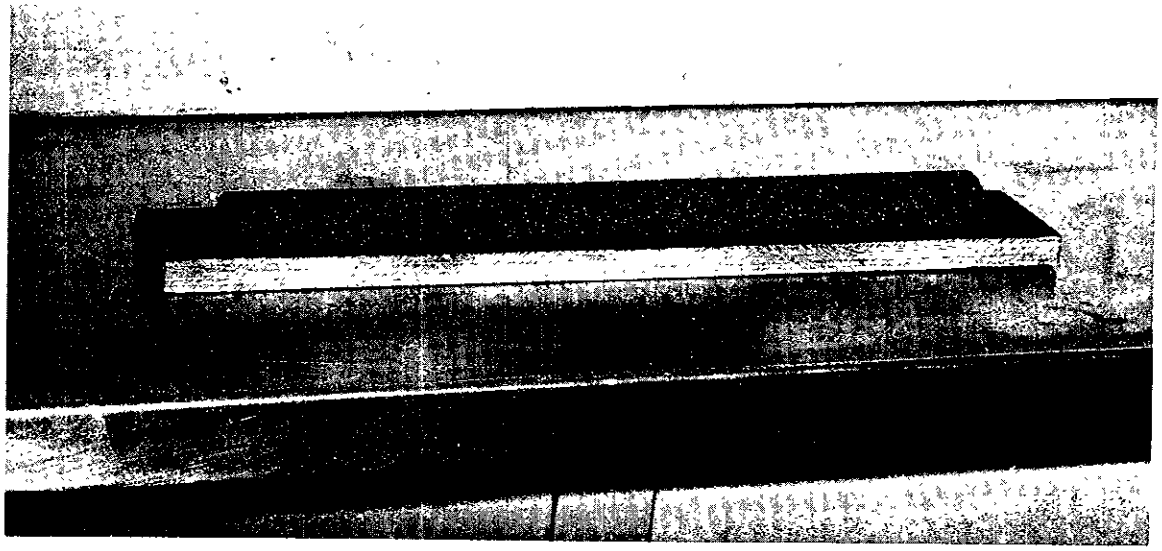


Figure III
Sample After Removing Paper Liner

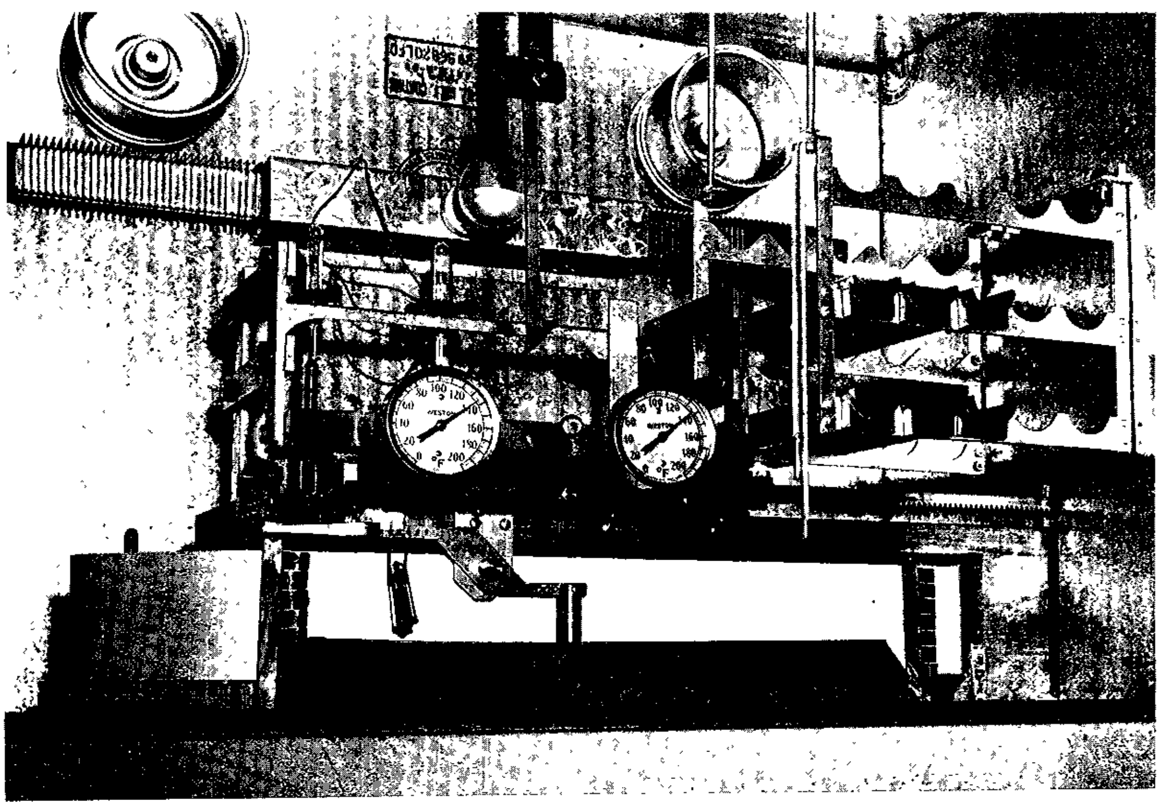


Figure IV
Mold Containing Sample In Place In
Cohesiograph Ready For Testing

December 1962

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

PREPARATION OF 2% ASPHALT OTTAWA SAND MIX BY THE SOLVENT METHOD

A. Procedure

1. Weigh 300 gms. of Ottawa sand, (No. 20 to No. 30), (ASTM C 190), in a 6" diameter, 2" high pan.

2. Accurately weigh 6.1 gms. of asphalt in a 100 ml beaker, add 10 ml of carbon disulfide, cover with a watch glass and allow to stand for one hour.

3. After the asphalt has dissolved, pour the solution over the Ottawa sand.

4. Immediately rinse the beaker with an additional 10 ml of carbon disulfide and pour the contents over the sand.

5. Immediately mix with a trowel until the mixture becomes stiff and hard to manipulate. If the material is mixed too long the asphalt will start to strip from the sand.

6. Set the pan containing the mixture in a ventilated hood at room temperature for twenty hours, and then place in an oven maintained at 140°F for an additional two hours.

METHOD OF TEST FOR DETERMINING THE EFFECT OF HEAT AND AIR ON A MOVING FILM OF ASPHALT (ROLLING THIN FILM OVEN TEST)

Scope

This test is used to measure the effect of heat and air on a moving film of semi-solid asphaltic materials. The effects of this treatment are determined from measurements of the properties of the asphalt before and after the test.

Summary

A moving film of asphaltic material is heated in an oven for 75 minutes at 325°F (163°C). The amount of hardening is determined from physical tests. An optional procedure is also provided for determining the loss in weight.

A. Apparatus[1]

1. Oven—This shall be a double-walled electrically heated convection type. Its inside dimensions are: height 15″ (38.1 cm); width 19″ (48.3 cm) and depth (with door closed) 17½″ ± ½″ (44.5 ± 1.3 cm). The door shall contain a symmetrically located window with dimensions of 12″ (30.5 cm) to 13″ (33.0 cm) wide by 8″ (20.3 cm) to 9″ (22.9 cm) high. The window shall contain two sheets of heat resistant glass separated by an air space. The window should permit an unobstructed view of the interior of the oven. The top of the upper heating element shall be 1 ± ⅛″ (2.5 ± 0.3 cm) below the oven floor. The oven shall be vented top and bottom. The bottom vents shall be located symmetrically to supply incoming air around the heating elements. They shall have an open area of 2.31 ± 0.11 sq. in. (15.0 ± 0.7 sq. cm). The top vents shall be symmetrically arranged in the upper part of the oven and have an open area of 1.45 ± 0.07 sq. in. (9.3 ± 0.45 sq. cm). The oven shall be equipped with a proportional control thermostat capable of maintaining 325°F (163°C) temperature within ± 1.0°F (± 0.5°C). The sensing element of the thermostat shall be placed immediately adjacent to the thermometer bulb. The heating controls shall be capable of bringing the fully loaded oven back to the test temperature within a 10 minute period after insertion of the samples in a preheated oven.

The oven shall be provided with a 12″ (30.5 cm) diameter vertical circular carriage—see Figure I for details. This carriage shall be provided with suitable openings and clips for firmly holding eight glass containers—see Figure II—in a horizontal position. The vertical carriage shall be mechanically driven through a ¾″ (1.9 cm) diameter shaft at a speed of 15 ± 0.2 RPM.

The oven shall be equipped with an air jet positioned to blow heated air into each bottle at its lowest point of travel. The air jet shall have an outlet orifice 0.04″ (1.016 mm) in diameter—No. 60 drill—connected to a 25′ (7.6 meter) length of 5⁄16″ (0.8 cm) O.D. copper tubing. This tubing shall be coiled to lie flat on the bottom of the oven and lead to a source of fresh, dried, dust-free regulated air.

NOTE 1—Activated Silica Gel treated with an indicator is a satisfactory desiccant for the dried air.

2. Flowmeter—The flowmeter may be any suitable type capable of accurately measuring the air flow at a rate of 4000 ml. per minute at the outlet of the copper tube.

3. Thermometer—This shall be an ATSM High Softening Point Thermometer conforming to the requirements 16F or 16C as prescribed in the Standard Specifications for ASTM Thermometer (ASTM Designation: E-1).

4. Container—The container in which the sample is to be tested shall be of heat resistant glass conforming to the dimensions shown in Figure II.

B. Preparation of Oven

1. Position the air outlet orifice so that it is ¼″ (0.635 cm) from the opening of the glass container. The orifice shall also be so positioned that the jet blows horizontally into the central arc of the opening of the circling glass container.

2. Position the thermometer specified in A-3 so that the end of the bulb of the thermometer is 1″ (2.54 cm) above the center of the shaft holding the revolving carriage. The stem of the thermometer shall be ¼″ (0.635 cm) from the opening of the glass containers.

3. Level the oven so that the horizontal axes of the glass containers when in position in the carriage are level.

4. Preheat the oven for a minimum of 16 hours prior to testing with the controls on the setting which will be used during the operation of the oven. The control thermostat shall be adjusted so that when the oven is fully loaded and the air is on, it will return to 325°F ± 1.0°F (163°C ± 0.5°C) within the 10 minute warm-up period.

C. Procedure

1. The sample as received shall be free of water. Heat the sample in its container with a loosely fitted cover in an oven not to exceed 325°F (163°C) for the minimum time necessary to insure that the sample is completely fluid. Manually stir the sample but avoid incorporating air bubbles.

2. Pour 35 ± 0.5 grams of the sample into each of the required glass containers providing sufficient material for characterizing tests which are to be run on the residue.

3. When the loss in weight is not required, allow the container to cool to approximately room temperature before placing in the oven as directed in Section C-4. When the quantitative value of the loss is desired, two separate bottles should be used for the determination.

[1] Complete equipment may be obtained from James Cox and Sons Inc., Colfax, California.

Cool the bottles for the test to room temperature and weigh each bottle separately to the nearest 0.001g.

NOTE 2—Do not use the residue from loss in weight determination for other tests.

4. With the oven at operating temperature, arrange the containers holding the asphalt in the carriage so that the carriage is balanced. Fill any unused spaces in the carriage with empty containers. Close the door and rotate the carriage assembly at a rate of 15 ± 0.2 RPM. Start the air flow at a set rate of 4000 ± 200 ml. per minute. Maintain the samples in the oven with the air flowing and the carriage rotating for 85 minutes. The test temperature 325°F ± 1°F (163°C ± 0.5°C) shall be reached within the first 10 minutes —otherwise discontinue the test. At the conclusion of the testing period remove the containers from the oven. If the loss is not being determined, proceed in accordance with Section C-5. For the glass containers on which the loss is being determined, cool to room temperature in a desiccator, then weigh to the nearest 0.001 g. and calculate the loss on the basis of the asphalt in the container. Discard the residue.

5. Immediately pour all of the residue, without scraping, from each bottle into a container large enough so that when all of it is collected the container is not over 75 percent full. Do not let the moving film bottles cool nor should the bottles be reheated to obtain more residue. Proceed as described in Section C-6.

6. Test the residue within 24 hours of performing the moving film test.

D. Report

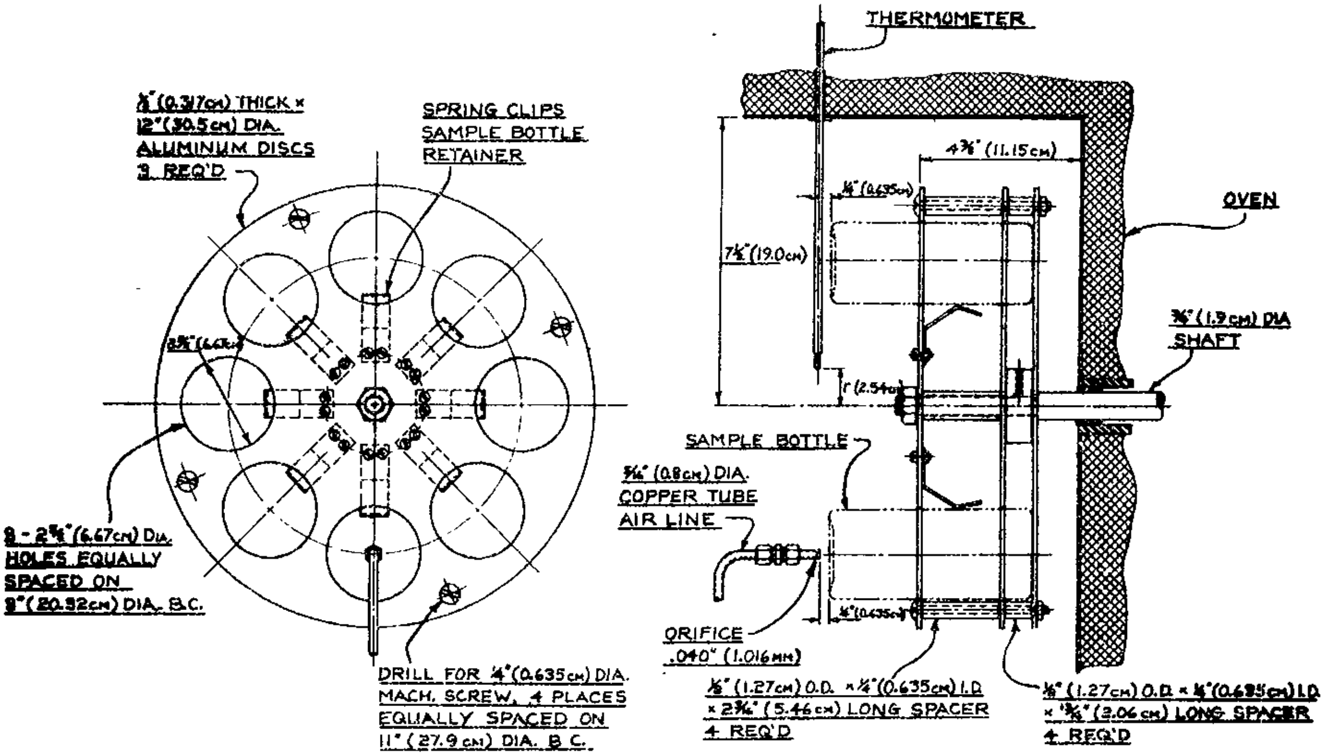
1. Report the results from the moving film test in terms of the physical changes in the asphalt brought about by this method. These values are obtained by performing appropriate tests on the asphalt before and after the moving film oven cycle.

REFERENCES

A California Test

End of Text on 346-D

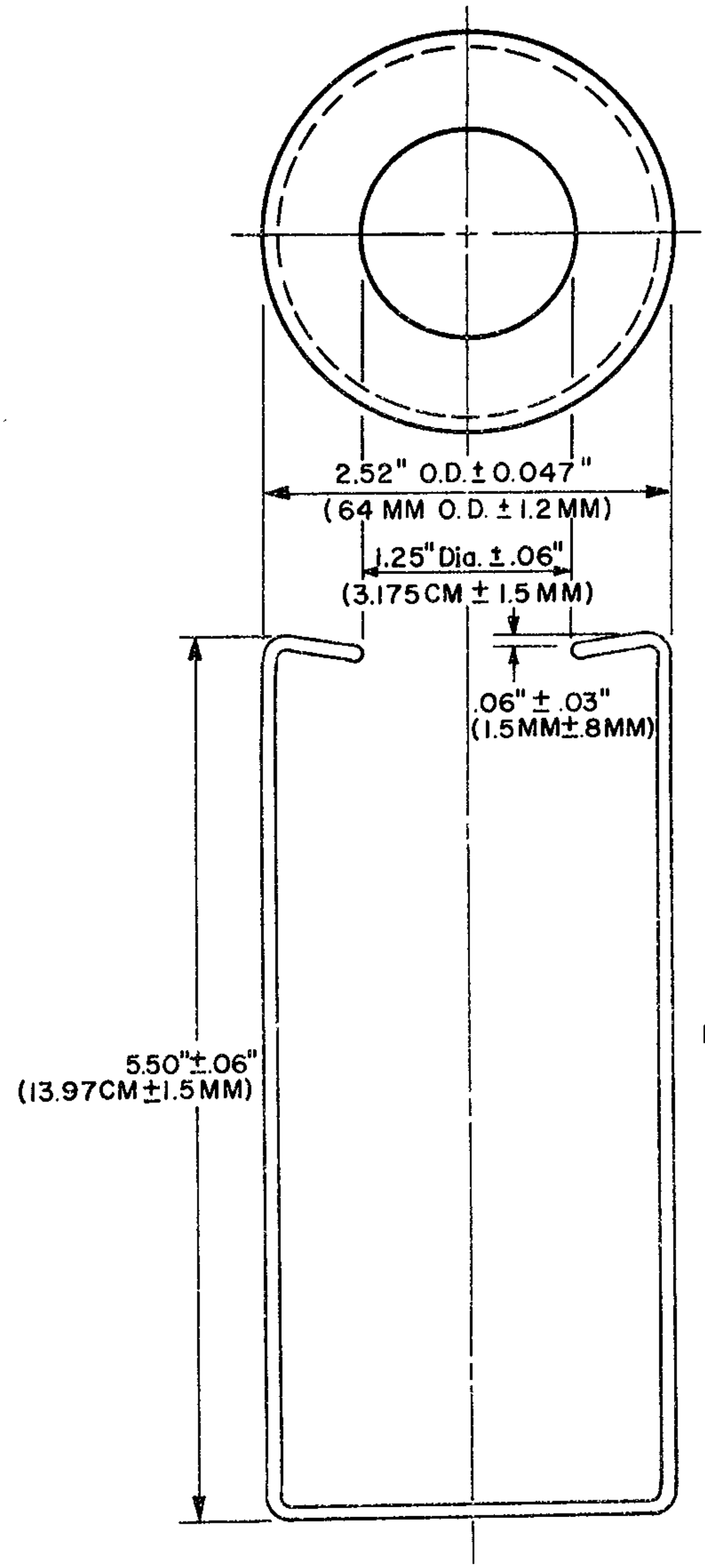
ASTM Designation: E-1



FRONT VIEW SIDE VIEW

CIRCULAR METAL CARRIAGE

FIGURE 1



MATERIAL–

PYREX BRAND TUBING
STANDARD WALL
64 MM O.D.
WALL THICKNESS
2.4 MM ± .3 MM

SAMPLE BOTTLE

FIGURE II

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

TENTATIVE METHOD OF TEST FOR DETERMINING THE EFFECT OF HEAT AND AIR ON PAVING GRADE ASPHALTS IN THIN FILMS

Scope

This method of test is intended to measure the effect of heat and air on paving grade asphalts. The test provides a relative measure of the durability during pavement service life.

Outline of Method

Twenty micron films are prepared, using the residue from the Rolling Thin Film Test, California Test Method 346-A. The films are heated in air at a temperature of 210F for twenty-four hours. The viscosity at two different shear rates and the ductility are determined on the residue remaining after the heating period.

A. Apparatus

1. Oven - The oven shall be rectangular in form with double walls and heated with electricity. It shall have controls capable of maintaining a temperature of 210 ± 2F. Its interior dimensions shall be as follows: Height, exclusive of space occupied by the heating element, not less than 15 inches; width, not less than 19 inches; and depth, not less than 18 inches. The oven shall have in front a tightly fitting hinged door which shall provide a clear opening substantially of the interior height and width of the oven. The door shall have a symmetrically placed double window with a clear opening of 8" x 8".

The oven shall be adequately ventilated by convection currents of air and for this purpose the oven shall be provided with openings for the entrance of air and the exit of the heated air vapors. Openings for the entrance of air in the bottom of the oven shall be symmetrically arranged and so placed that incoming air will circulate around the heating elements; the openings shall have a total area of not less than 2.20 sq. in. nor more than 2.42 sq. in.. Openings for the exit of heated air and vapors in the top of the oven shall be symmetrically arranged and have a total area of not less than 1.38 sq. in. nor more than 1.52 sq. in..

The oven shall be provided with a perforated aluminum shelf, 16" in diameter. A recommended form of shelf is shown in Figure I. This shelf shall be placed in the center of the oven with respect to all dimensions of the interior of the oven, shall be suspended by a vertical shaft and provided with mechanical means for rotating it at the rate of 5 to 6 r.p.m.

2. Thermometer - The thermometer shall be a specially fabricated mercury thermometer with the following specifications: Range, 205F to 215F; Length, 6"; Immersion, Total; Divisions, 1/2°F; Scale error, shall not exceed 1/2°F.

3. Weathering Plates - The plates shall be of polished Pyrex plate glass, 50±0.1 mm by 50±0.1 mm and 6±0.1 mm thick, in matching pairs.

4. Infrared Lamp Hotplate, operated from a variable transformer.

5. Analytical Balance - Accurate to 0.1 mg.

B. Materials

1. Razor Blade - Single edge type for scraping excess asphalt from weathering plates.

2. Paper Tissue - Paper towels and "Kleenex" or equal.

3. Solvents - Benzene.

C. Preparation of Sample

Carefully heat the sample in an oven, maintained at a temperature not over 325F, until the material is in a fluid condition. After melting, thoroughly stir the sample until it is homogeneous and free from air bubbles.

D. Procedure

1. Place a drop of the stirred sample on one of a pair of weathering plates, which have previously been cleaned with benzene and weighed to the nearest 0.1 mg. Cover the asphalt with the second plate and place the plates on the infrared hot plate to melt the asphalt thoroughly.

2. As the asphalt softens, press and work the plates together with a circular motion. Continue as necessary to form a uniform film of asphalt as judged by viewing in the transmitted light from the infrared lamps. Holding the plates up to a fluorescent or incandescent light works well also. By experience an operator may judge the approximate thickness by the color of the asphalt film.

3. After forming the film, allow the plates to cool. Scrape any excess asphalt from the edges of the plates with a razor blade and wipe the plates clean with paper tissue (such as Kleenex) or a cloth wetted with benzene.

4. Weigh on an analytical balance to the nearest 0.1 mg. to determine the weight of the film. The film thickness must be 36.0 to 44.0 microns.

5. Calculate the film thickness from the following equation:

$$\text{Film thickness, microns} = \frac{10^4 W}{lwd}$$

Where: d = density of asphalt, grams per cubic centimeter. (for most asphalts assume d = 1)

l,w = length and width of the glass plate in centimeters.

W = Weight of asphalt film in grams.

6. Warm the plates with the infrared lamp to soften the asphalt and then separate them by sliding the upper plate from the lower one. Place the separated plates on a piece of paper toweling 2-1/2" x 5-1/2" and then place on the revolving shelf of the oven, specified in A-1, maintained at 210±2F. (See Note 2) Figure I illustrates placement of plates, on paper towel, on shelf in oven.

7. Determine the temperature by means of the specified thermometer, A-2, supported from the shaft of the circular shelf in a vertical position at a point equi-distant from the center and the outer edge of the shelf with the bottom of the bulb of the thermometer 1/4 inch above the revolving shelf.

8. Cure the samples for a period of 24 hours with the oven shelf revolving at 5 to 6 r.p.m. during the entire curing period.

9. At the conclusion of the prescribed curing period, remove the plates from the oven, cool to room temperature, and scrape the asphalt off of the plates with a razor blade, which has previously been cleaned with benzene and dried.

10. Transfer part of the material to a clean, weighed viscosity plate and prepare for viscosity measurement as described in "Method of Test for Determination of Viscosity of Bituminous Materials by Means of the Microviscometer", Calif. No. 348-A.

11. Prepare 3 micro-ductility molds from the balance of the material as described in "Method of Test for Micro-Ductility of Bituminous Materials", Calif. No. 349-A.

E. Calculation and Report

1. Perform required calculations as described in "Method of Test for Determination of Absolute Viscosity of Bituminous Materials by Means of the Microviscometer", Calif. No. 348-A.

2. Plot log shear rate versus log viscosity and draw a straight line through the points. From this line read the viscosity at a shear rate of 0.05 sec^{-1} and 0.001 sec^{-1}. Report the viscosities in megapoises for these shear rates.

3. Determine the average value for the three micro-ductility results as specified in Calif. No. 349-A.

F. Notes

1. A suitable oven is the "Blue Line" oven Model OV-18C available from Blue M Electric Company. This oven does not contain the window in the door, but may be so altered.

2. A suitable paper to place the plates on in the oven may be made by dividing a standard 5-1/2" x 10" folded paper hand towel into four equal parts.

REVOLVING SHELF FOR DURABILITY TEST OVEN

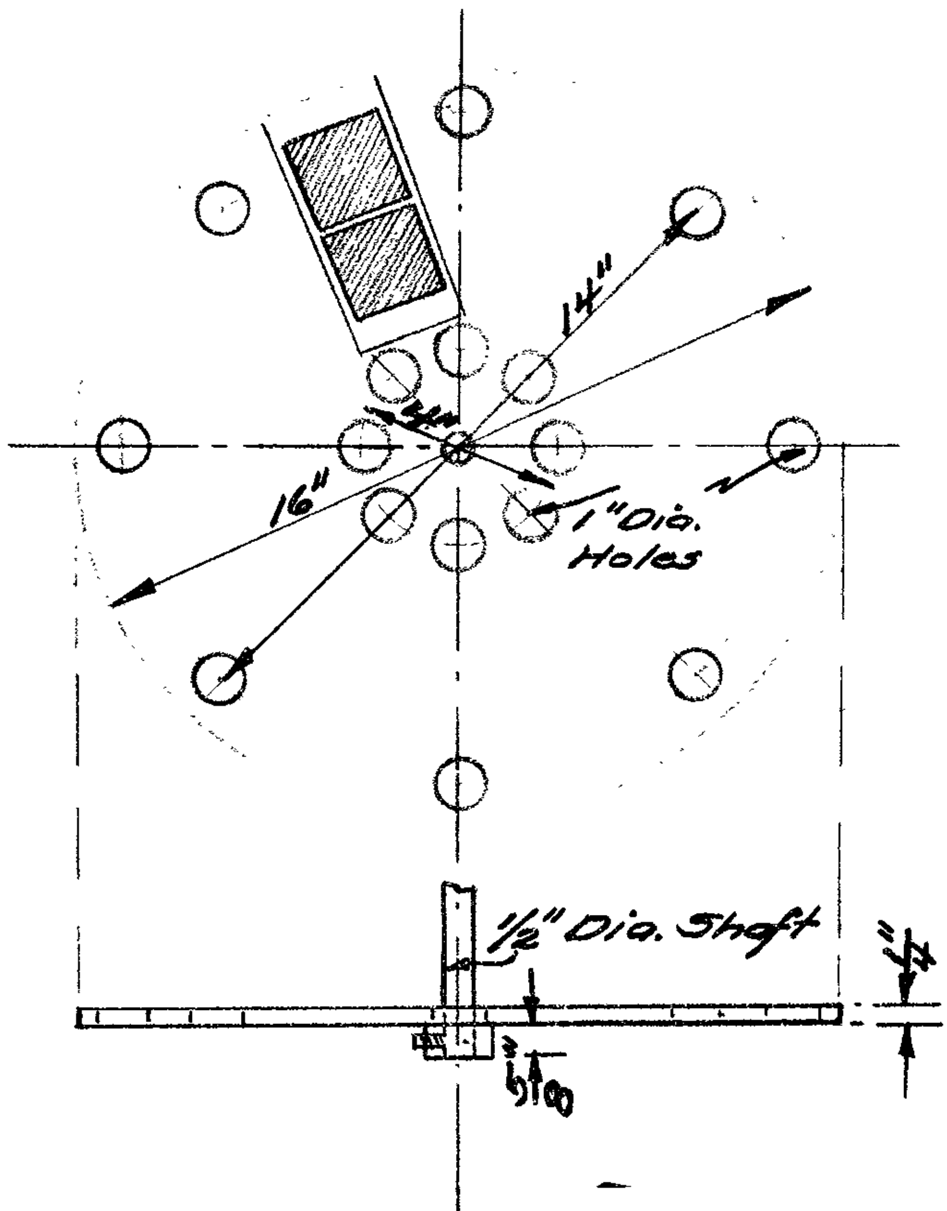


FIGURE I

Test Method No. Calif. 348-A
November, 1962
(4 pages)

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

TENTATIVE METHOD OF TEST FOR DETERMINATION OF VISCOSITY OF BITUMINOUS MATERIALS BY MEANS OF THE MICROVISCOMETER

Scope

This method of test is intended to measure the viscosity of bituminous materials by means of a sliding-plate microviscometer.

A. Apparatus

1. Microviscometer - Sliding plate type, Shell Development Company design.

2. Viscosity Plates Polished Pyrex plate glass, 20±0.1 mm by 30±0.1 mm by 6±0.1 mm thick, in matching pairs.

3. Viscosity Water Bath - The water bath shall maintain the temperature to within ±0.2 F. The depth of water above the perforated shelf must be at least five inches and should be approximately seven inches.

4. Viscosity Bath Thermometer - A total immersion thermometer of suitable range, graduated to 0.1 or 0.2 F (0.05 or 0.1 C) and conforming to the general requirements of ASTM Specification E-1 shall be used. ASTM Saybolt Viscosity thermometers and ASTM Kinematic Viscosity thermometers are suitable.

5. Infrared Lamp Heating Unit - The heating unit shall be operated from a variable transformer.

6. Analytical Balance - Accurate to 0.1 mg.

B. Preparation of Sample

Carefully heat the sample in an oven, maintained at a temperature not over 325 F until the material is in a fluid condition. After melting thoroughly stir the sample until it is homogeneous and free from air bubbles.

C. Procedure

1. Specimen Preparation. Prepare specimens for viscosity measurements by the following method:

a. Place a drop of the melted sample on one of a pair of viscosity plates which have previously been cleaned with benzene, dried with paper tissue and weighed to the nearest 0.1 mg.

b. Cover the asphalt with the second plate and use the infrared lamp to melt the asphalt thoroughly. As the asphalt softens, press and work the plates together.

c. Repeat as necessary to form a uniform layer of asphalt as judged by viewing in transmitted light. (Recommended film thicknesses are given in Table 1.)

d. After forming the film allow the plates to cool. Scrape any excess asphalt from the edges of the plates with a razor blade and wipe the plates with a cloth dipped in benzene. Weigh on an analytical balance to the nearest 0.1 mg. to determine the thickness.

2. Calculate the film thickness by means of the following equation:

$$\text{Film Thickness, microns} = \frac{10^4 W}{lwd} = \frac{10^4 W}{6} = 1667 W$$

Where:

d = density of the sample, grams per cubic centimeter (for most asphalts assume d=1).

l,w = length and width of the glass plate, centimeters (for the plates obtained from the recommended sources, l = 2.00 and w = 3.00).

W = weight of sample, grams.

3. **Viscosity Measurement.**

a. Allow the specimen loaded between the viscometer plates to cool in air for 60 to 70 minutes.

b. Then place the viscometer plates in the viscometer which has been standing in the water bath. After loading the plates in the viscometer, allow 2 to 5 minutes for the sample to come to the bath temperature.

c. Clean the contact flag and micrometer point by rubbing lightly with Crocus cloth (See Note 1).

d. Position the flag beneath the micrometer and move the micrometer, by driving the motor, until it makes contact with the flag.

e. Apply the load by adding weights to the weight holder on the beam and measure the displacement either by reading the micrometer as a function of time or by recording the displacement graphically on a milli-volt recorder (See Note 2).

f. By the use of four different loads (See Note 3), determine the viscosity of each sample at four shear rates which bracket the desired shear rate (See Note 4). Normally 0.05 sec^{-1} will be the standard shear rate at temperatures above 50 F. Below this temperature, 0.001 sec^{-1} will be used; suggested loads are given in Table 1. It is usually possible to make a single viscosity determination with 0.1 mm movement; therefore, a total movement of 0.5 mm should be sufficient for four shear rates. In no case should the total movement exceed 1.50 mm as this changes the effective area of the sample.

Note 1: In routine operation, cleaning of the contact flag and micrometer is required only about once a day.

Note 2: More detailed instructions for measuring the viscosity are given in the instruction manual supplied with the viscometer.

Note 3: The initial load should be the largest load in order to nullify the yield point.

Note 4: The largest weight used should be about six times as large as the smallest.

Table 1

SUGGESTED FILM THICKNESSES AND LOADINGS FOR DIFFERENT GRADES OF ASPHALT

PENE-TRATION AT 77 F	MEASUREMENTS AT 77 F		MEASUREMENTS AT 39.2 F	
	RANGE OF FILM THICKNESS, MICRONS	RANGE OF LOADS, GRAMS	RANGE OF FILM THICKNESS, MICRONS	RANGE OF LOADS, GRAMS
200-300	10-100	20-200	50-200	100-1000
120-150	10-100	50-500	50-200	500-5000
85-100	10-100	100-1000	50-200	1000-10,000
60-70	10-100	300-3000	50-200	2000-10,000
40-50	.10-100	500-5000	100-400	2000-10,000

D. Calculation and Report

1. Calculate the shearing stress for each load and the shear rate for the resulting plate movement from the following formulas:

$$\text{Shearing stress (dynes/cm}^2) = \frac{\text{Wt. applied (g) x 980}}{\text{Area of Plate (cm}^2)}$$

$$= 163.3 \text{ x Wt. applied (g)}$$

$$\text{Shear rate (sec}^{-1}) = \frac{\text{Plate Movement (microns)}}{\text{Time (sec) x Film Thickness (microns)}}$$

Calculate the viscosity for each load from the following equation:

$$\text{Viscosity (poises)} = \frac{\text{Shearing Stress (dynes/cm}^2)}{\text{Shear Rate (sec}^{-1})}$$

Plot log shear rate versus log viscosity and draw the best straight line through the points. From this line read the viscosity at the desired shear rate.

2. Report the viscosity in megaposes at the specified temperature and shear rate.

Test Method No. Calif. 349-A
November, 1962
(6 pages)

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

TENTATIVE METHOD OF TEST FOR MICRO-DUCTILITY OF BITUMINOUS MATERIALS

Scope

This test method provides a means of testing small quantities of bituminous materials for ductility.

A. Apparatus

1. Ductility machine, capable of pulling specimens at 1/2 cm/min. See Drawing No. D-507.

2. Ductility mold assembly - See Drawing No. B-507.

   a. Molds
   b. Mold Holders

3. Water bath with controls for regulating water temperature at 77±1F.

4. Metric scale with 1 mm divisions.

5. Hot plate.

6. Pick (made by flattening one end of a 4" length of welding rod). See Drawing No. B-507.

7. Magnifying Lamp - (a suitable lamp is the "Luxo Magnifying Lamp," Model LFM-1).

B. Preparation of Material

If the material to be tested is in a container it should be heated and stirred thoroughly before removing a test specimen.

C. Procedure

1. Place assembled molds (less plugs) on a hot plate previously adjusted to about 250 to 300 F surface temperature, a temperature that will liquify the asphalt to be tested. Do not heat plugs.

2. Secure about 0.05 grams of the material to be tested with the pick and transfer to the center of the large end of the plug, see Figure 1. Remove mold from the hot plate with a clamp, being careful that the halves remain tight together. Screw plug with asphalt into mold slowly, noting that the asphalt flows up through the orifice and fills the cone, see Figure 1.

3. Screw second plug into place. Do not tighten either plug excessively, to do so tends to separate the mold.

4. Let mold with clamp set in air at room temperature for 15 minutes minimum.

5. Place mold with clamp in water bath maintained at the desired testing temperature for at least 10 minutes before testing.

6. Transfer mold to ductility machine using clamp.

7. Turn mold so that slot will be in a convenient position to view specimen at start of test.

8. Tighten thumb screws.

9. Bring the magnifying lamp into position so that as the mold halves begin to separate, viewing of the asphalt thread is possible.

10. As mold halves start to separate force water with an eye dropper through the molds at the point of separation to force air bubbles off the test specimen. When asphalt thread breaks, shut off motor.

11. Remove apparatus from water bath and measure length of thread to nearest millimeter.

D. Calculation and Report

1. Determine the average value for the three micro-ductility results as specified below:

Group 1 - All three readings on any sample are within 0 through 30 mm. Average all three results for any sample, if the three readings are within a range of 4 mm. If greater than 4 mm, average the two highest results within this range, and discard the third reading. If any two of the three results are not within the 4 mm range, then the test shall be repeated.

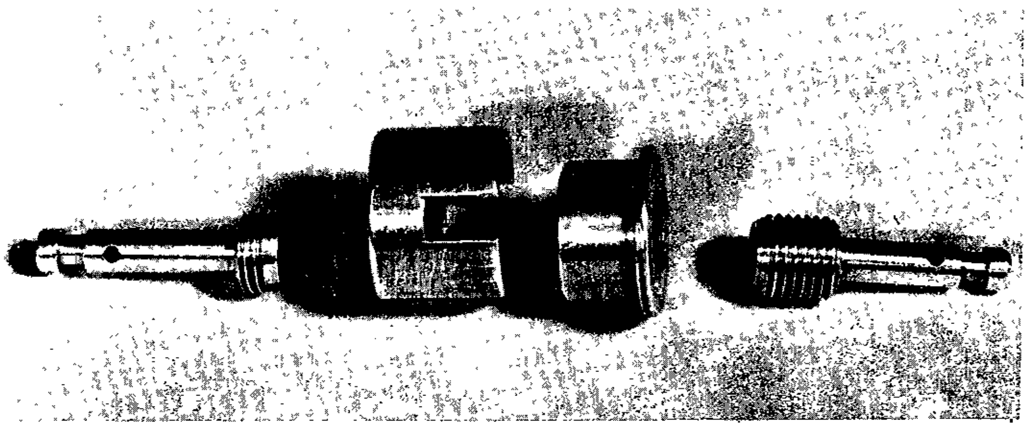
Group 2 - All readings on all samples are above 30+ mm. Average the two readings that are nearest to each other and discard the third.

If one reading is above 30 mm. and two readings are below, the requirements for Group 1 shall apply. If two readings are above 30 mm, and one below, the requirements for Group 2 shall apply.

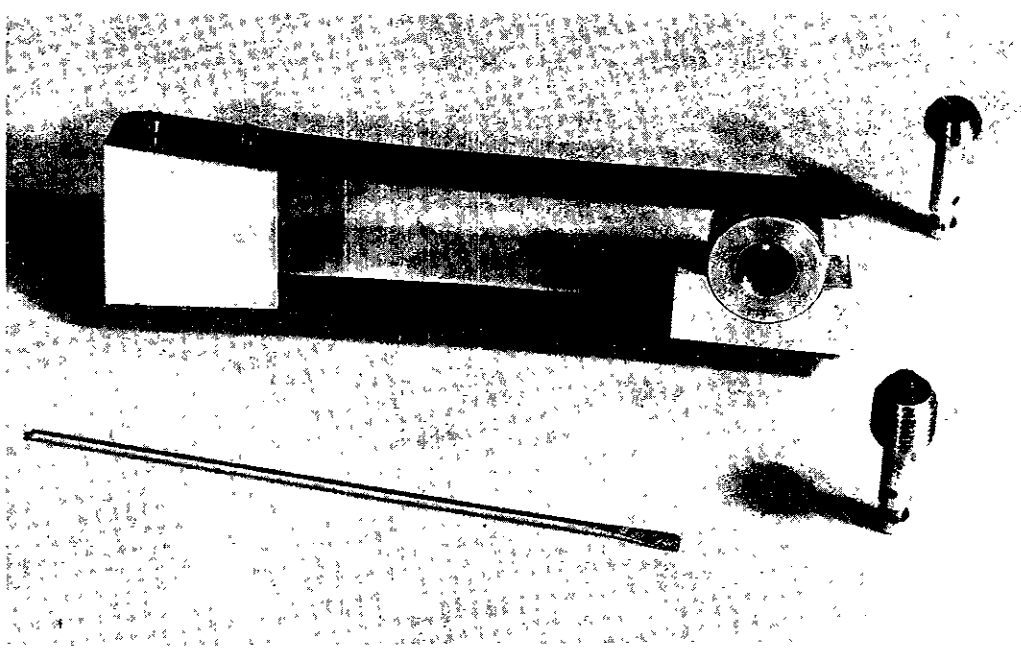
E. Notes

1. For cleaning, plugs may be unscrewed from molds easily by inserting a piece of 1/16" diameter welding rod through hole in stem to give leverage.

2. In handling molds with clamps be sure that both halves are securely gripped.

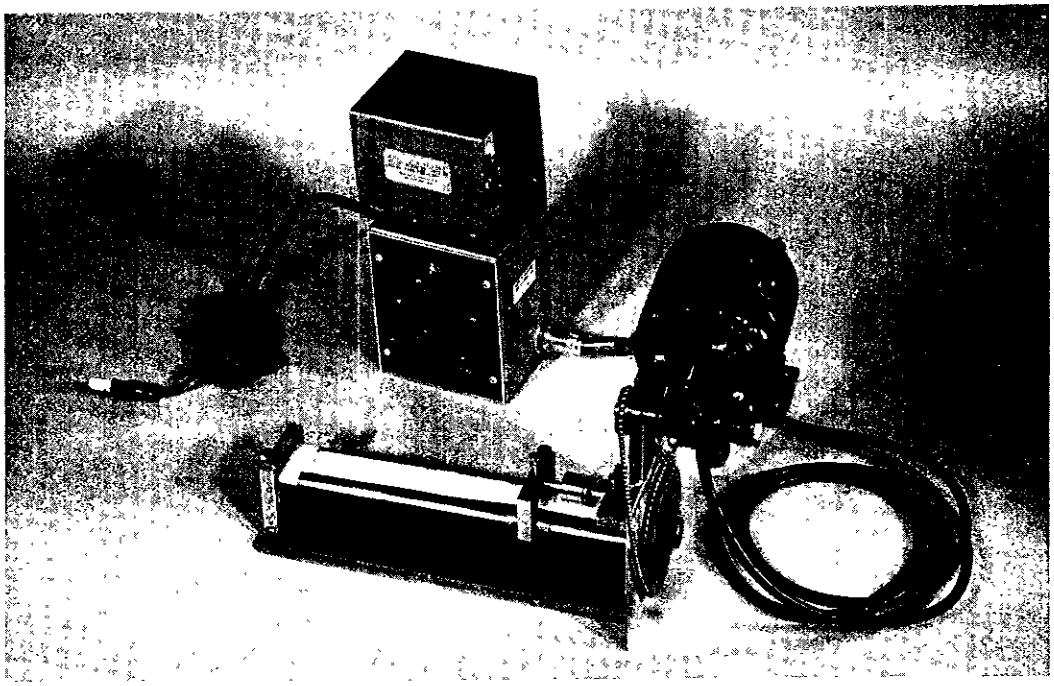


Ductility Mold and Plugs

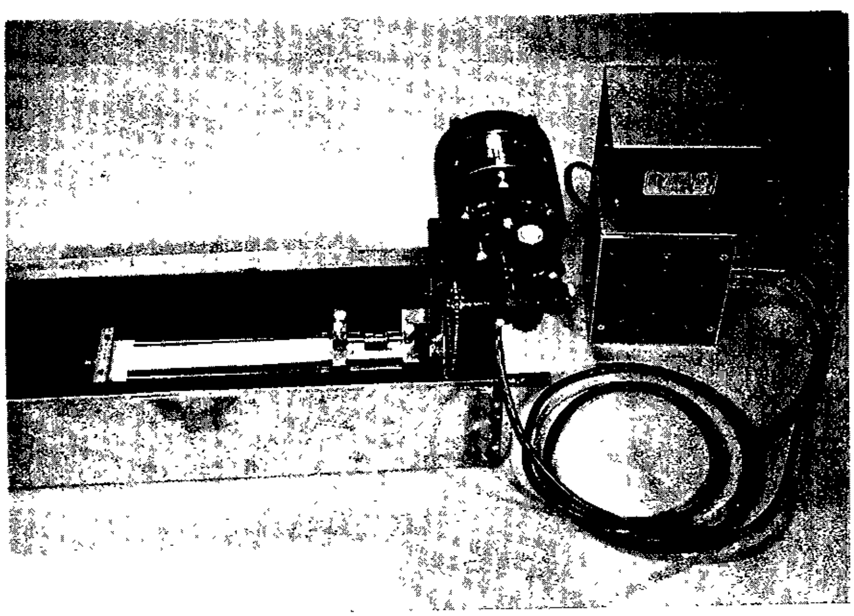


Ductility Mold
Asphalt for Specimen is on Plug in Foreground
Note Pick for Transfer of Asphalt to Plug

Figure I

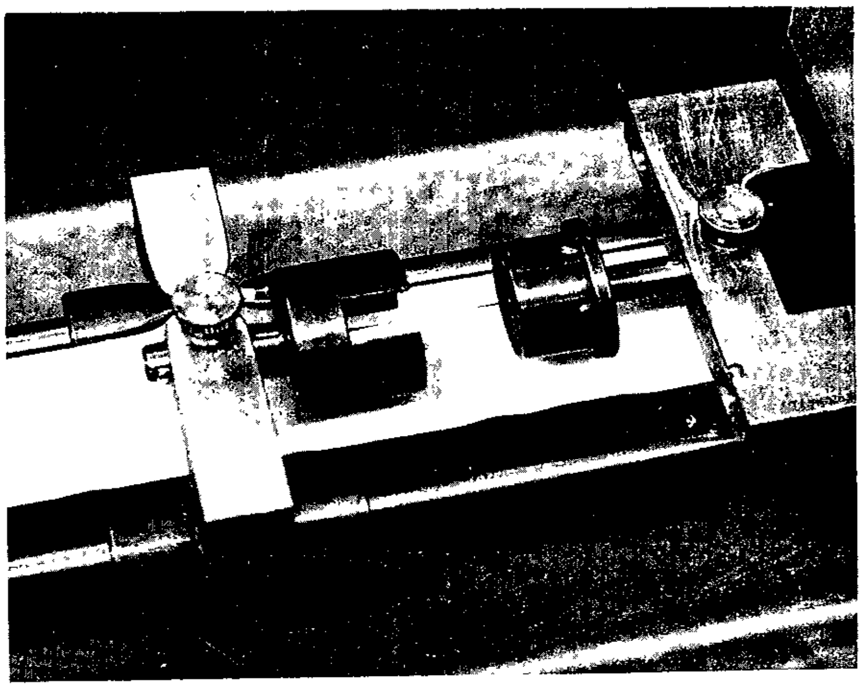


Ductility Test Apparatus



Ductility Test Apparatus
in Water Bath

Figure II



Ductility Test in Operation
Note Asphalt Thread

Figure III