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The effect of filler on asphalt cement mastics

Dah-yinn Lee
Iowa State University

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THE EFFECT OF FILLER ON ASPHALT CEMENT MASTICS

by

Dah-yinn Lee

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INTRODUCTION

The problems of the asphalt paving technologists today are complicated by radically changing conditions. It is readily apparent that today's pavements are being subjected to constantly increasing heavy wheel loads and constantly increasing traffic density. And it can be safely assumed that this trend will continue. On today's airfields, especially military airfields, the situation is even more drastic; pavement loading and tire pressures have increased in proportions undreamed of a quarter of a century ago.

For pavements to serve satisfactorily under these increasingly severe conditions, for both present and future developments, it is clear that the asphalt industry and the paving engineers charged with the design and construction of pavement structures must search continually for better and more exact procedures in pavement design, for improvement in construction equipment and practices, and also for better understanding, therefore, more intelligent use, of old as well as new construction materials.

Because an asphalt paving mixture is a composite material, changes in various components produce different engineering properties. When all the ingredients of the mixture, which consists of coarse and fine aggregates, mineral filler, and asphalt cement, are mixed in proper balance, the theoretical end result is an ideal paving mixture.

Although research work has been done on the effects of coarse aggregate and fine aggregate, the effects, functions and potentialities of the filler are not well defined. In spite of the small proportion it represents in the mix, it has been proved of critical importance in the performance of the finished pavement.

The use of mineral dust in asphalt paving mixtures, as well as in a variety of special applications of asphalt, long has been common practice. Natural asphalts, used by older civilizations and still used extensively in some European countries, usually contain fairly large quantities of mineral dust. On the other hand, petroleum asphalts, used almost exclusively in the United States are products of the crude oil refining process and are free from such mineral matter. Thus the engineer has to select the type and quantity of mineral dust best suited for his purpose. This selection of mineral dust has been a matter of empirical practice.

Mineral fillers are generally defined as fine inert mineral dusts, a high proportion (a minimum of 65 percent by weight by A.S.T.M. and A.A.S.H.O. specifications) of which will pass the No. 200 sieve. A large number of materials are used as mineral fillers, including limestone dust, volcanic ash, silt, powdered shale, portland cement, mineral sludges, hydrated lime, rock flour, diatomaceous earth, and fly ash (32, 83, 89).

The mineral filler can greatly affect the properties of a mixture such as strength, plasticity, voids, resistance to action of water, and the resistance to the forces of weathering. The proper use of filler can improve the asphalt paving mixture through increased density, stability, durability, and skid resistance. On the other hand, excessive quantity of filler tends to increase brittleness and proclivity to cracking, and deficiency of filler tends to increase void content, lower stability, and soften the mix which leads to shoving, and rutting of the pavement.

Over years of experience, asphalt paving technologists have generally agreed on what are satisfactory fillers, what are unsatisfactory fillers, as well as the optimum filler content that should be used in order to get a good paving mixture of certain types. However, in order to design the mix more scientifically to meet constantly increasing traffic loads, the proper understanding of the effect of filler on the asphalt binder, the functions to be served by the mineral filler, and the appropriate selection of filler type and quantity to achieve these functions are essential.

Some paving technologists believe that the function of filler is to change the rheological properties of the binder. The filler and asphalt combine to form a mastic which acts to fill voids and also bind aggregate particles together to a dense mass. (12, 21, 32, 49, 50)

Others consider the principal function to be the filling of the void spaces between the coarser aggregate fractions of the mix. They suggest that mineral filler can be used to replace a certain amount of asphalt in the mix, and that the gradation of filler is of utmost importance (1, 46, 48, 78, 87).

Still others (33, 60, 70) approach the subject from a surface area concept, concluding that the large surface area of mineral dust will result in an increased demand for asphalt since there is more available surface requiring asphalt coating. Thus the fineness of the filler is of utmost importance. It can be readily seen that the latter two viewpoints are in direct conflict, and it appears that neither is entirely valid.

There are also others who consider the filler having a dual function. While the extremely fine particles may be located in the asphalt films that coat the coarser aggregate particles, the coarser filler may be viewed as part of the aggregate skelton of the pavement. Therefore the extremely fine portion of the filler may alter and have an appreciable effect on the apparent viscosity characteristics of the asphalt, and the coarser portion of the filler may perform the same function as the coarse and fine aggregate in resisting stresses imposed on the pavement (33, 40, 54, 70).

Further, as pointed out by Professor Csanyi (21), fillers may function and behave in many different ways depending on mixing procedures and the properties of the filler used in preparing the mix. In mixes prepared in the conventional manner, fillers may serve predominantly to fill voids, while mixes produced by special processes such as the foamed asphalt (19), impact (22) and cooking processes, (18) virtually all of the filler may be used in the development of a mastic which serves to fill the voids between aggregate particles and as a binder to bind aggregate particles together, thereby yielding mixes of higher density, stability, and toughness.

It is therefore clear that more extensive basic studies on the use of mineral fillers in the bituminous mixes are needed. These studies should provide information such as: What are the specific functions of the filler? How do the various types of filler react with the asphalt cement? What are the properties of the filler that are important with respect to the performance of the paving mixtures? How does the mineral filler content of the mix affect the balance of ingredients in the mix? It was with the goal of answering these questions that this investigation was undertaken.

PURPOSE AND SCOPE

It has long been recognized by the asphalt paving technologists that the addition of mineral filler in asphalt paving mixtures is essential to the better performance of the pavements. The incorporation of mineral powders in bituminous materials for other commercial applications, such as asphalt plastic, asphalt mastic, asphalt roofing, etc., has also become increasingly important. It is apparent that the filler must be able to change the characteristics of asphalt as well as the asphalt mix into a superior binder or mix possessing more desirable properties.

Though many investigators have studied the effects of fillers on the physical properties of a bituminous mix which could explain, to some extent, the favorable use of fillers in paving mixtures, most of the work has been directed toward the determination of the optimum quantity of filler or the determination of the best suitable type of filler for a specific mix. As a result, the definition of the filler has not yet been precisely defined, and the specific effects of the filler in a bituminous mastic has not been clearly understood.

Consequently a systematic study of the asphalt-filler system is warranted and desirable. Such study should aid in understanding the functions of the filler in a bituminous paving mix, in clarifying the advantages to be gained by the proper use of the mineral fillers, and in providing information for a more scientific design of asphaltic mixes.

Therefore, the object and scope of this investigation, sponsored by the Iowa Highway Commission, is directed toward four broad areas:

1. To determine the effects of fillers on the physical properties of various asphalt-filler systems containing:

    (a.) Varying proportions of filler

    (b.) Fillers of different natures

    (c.) Fillers of different sizes

    (d.) Asphalts of different grades

2. To relate these effects of the filler upon the asphaltic mastics prepared within 275 °F to 325 °F temperature range to the particle size, gradation, particle shape, as well as type of the filler.

3. To postulate the functions of fillers in the bituminous mix from these effects of the fillers on the asphalt-filler system.

4. To redefine the term mineral filler according to the specific function it performs in a bituminous mix.

LITERATURE REVIEW

Function of Fillers

The importance of using mineral fillers in asphaltic paving mixtures has been very well recognized. The specifications for fillers, though not all in agreement from state to state, have also been very well established. The practice, backed by experience, has been proved satisfactory. Unfortunately, the investigations concerning the functions and behavior of fillers have not been able to yield enough information.

The intention of using filler in asphalt mixes can be traced back to DeSmedt in 1870's, but until as late as 1893 there was still a question as to whether or not it was beneficial to add filler in the paving mixtures (60). In the early practices, only carbonate of lime was used (78), because it was thought that there was some chemical reaction between the bitumen and carbonate, but this idea was pretty well exploded by 1900, and paving experts generally believed at that time that a pulverized silica was as good as, if not better than, carbonate of lime.

In his book, "Street Pavements and Paving Materials" in 1900, Tillson (78) brought up the object of the powdered mineral matter as to fill the voids in the sand so as to make the total voids as small as possible and thus the exact quantity to be used in an asphaltic wearing surface mixture should be determined by the gradation of the sand.

Clifford Richardson (59, 60) was probably the first to describe the importance of filler. He believed that particles smaller than 0.05 mm were the most valuable particles, and suggested that a good filler should contain at least 60 percent by weight particles smaller than this size. He also proposed the dual function of filler as: (a.) Rendering the mixes higher density, and (b.) stiffen the asphalt cement. His view was shared by Spaulding and others (33, 40, 54, 70).

Satisfactory fillers recommended by Richardson included portland cement, ground limestone, ground shale and ground clay.

In 1913, Richardson (59) extended the function of filler to include making the asphalt cement less susceptible to changes in consistency caused by heat. Filler was defined as a part of the mineral filler with at least 75 percent passing # 200 sieve and at least 66 percent remaining suspended in water for 15 seconds. Acceptable fillers were extended to include ground traprock, marl and volcanic ash.

In 1915, Richardson presented "The Theory of the Perfect Sheet Asphalt Surface" (61), in which he stressed the importance of fine particle size and surface area of the filler, saying,

> We now understand the fact that an extended surface area in addition to providing for the use of a larger amount

> of bitumen exercises a still more important function, due to the greater surface energy developed by the larger surface area of a fine mixture over that of a coarse one and that, aside from the greater surface presented by a fine sand as compared to a coarse one, the presence of highly dispersed colloids with their extensive surface is necessary for the production of the most satisfactory surface.

This concept was shared later by many others (33, 86, 87) with regard to the function of the filler.

Spielmann and Hughes (74) agreed with Richardson's conception that the filler forms a colloidal suspension in bitumen and together fills the voids in the aggregate. In addition, they specified that the immediate effect of the admixture of filler to bitumen was to increase its adhesive powers, and raise its softening point and its general stability.

Besson (11) and Macnaughton (46) believed that dust was added not to reduce the total volume of voids, but to reduce the size of individual voids, and increase their number. Based on his experiences, Macnaughton further rejected Richardson's idea that the bitumen which a bitumen mixture would carry increases with an increase in surface area, (i.e. an increase in either filler content or the fineness of the filler), and that a well graded fine mixture with low voids would carry more bitumen than a coarser mixture with larger voids. He also suggested that the proper bitumen content in an asphalt paving mixture should be determined by the volume of the voids, and not by the surface area.

Miller and Traxler (49, 50, 84) believed that the function of filler was to change the consistency of the bitumen-filler system. The most important characteristics of filler were believed to be particle shape, texture, particle size and size distribution, and particular void space which governed the consistency or viscosity of the mixture.

The concept that the filler formed a colloidal suspension in the bitumen was supported by other investigators (12, 21).

The Corps of Engineers included filler study in a study of airfield pavements (87). This study is valuable because it included both laboratory stability analysis and test tracks in the field. It was their finding that filler was a void filling material, and in addition it reduced the size of the voids resulting in a mixture that was more dense, less permeable, and of finer texture than mixtures without filler. On the other hand excess filler might reduce flexibility and durability, and for that reason the best mixtures should contain as much asphalt and as little filler as possible. These findings were the same as believed by Agg (1) and Tillson (78).

The role of filler was described by Ridgen (63) in two categories. Low concentrations, filler was added to increase the stability of the bituminous surfacing by increasing the viscosity of the binder. High concentrations, filler-bitumen ratios greater than one by weight, were related to "optimum binder content." The condition for "optimum binder content" of any filler-binder system was that the voids in the compacted filler were just filled with binder, i.e. the system was one of maximum density which would give maximum resistance to deformation in tension.

Nevitt (54) had questioned the validity of the mastic concept on the basis of film thickness calculations and minus number 200 sieve particle sizes. On the other hand, Csanyi (21) had proved, from his observations and tests, that the basic assumption of the filler theory was not wholly valid when mixes were prepared by conventional means. He further suggested that fillers might function and behave in many different ways depending on mixing procedure and the properties of the filler used in preparing the mix.

In his review on mineral fillers, Tunnicliff (86) proposed a definition of filler in order to suggest the functions of filler in a bituminous mix. The proposed definition was:

> Filler is mineral material which is in colloidal suspension in the asphalt cement and which results in a cement with a stiffer consistency.

This proposal followed generally the same line of reasoning as those of Richardson (60), Bollen (12) Miller and Traxler (49, 50), Rigden (63) and Mitchell _et_ _al_. (51). He could also explain the mechanism, which accounted for the desirable characteristics of the filler-bitumen system such as stability and durability, by the surface energy of attraction, or adsorption, between the mineral particles and the bitumen. One question left unanswered: Which particles are colloidal in bitumen?

Since the term filler has not yet been precisely defined, the idea of the function of filler varies among asphalt paving technologists. However, it appears in a general way, that the filler used in bituminous mixes today functions in a dual role: while the coarser particles of the filler participate in producing contact points and filling voids, the finer particles of the filler may be suspended in the asphalt, changing the properties of the binder films. A number of factors, such as gradation of the rest of the mineral aggregate, methods of mixing, degree of compaction, thickness of binder films, viscosity of the asphalt, particle size distribution of the filler, relative proportions of asphalt and filler, and others may determine which function of filler will predominate.

Methods of Evaluation

Methods of evaluating fillers can be summed up into four categories:

1. Evaluation of filler by testing filler itself.
2. Evaluation of filler by testing filler-asphalt mastic system.
3. Evaluation of filler by testing the asphalt mixture.

4. Evaluation of filler by field traffic test.

Evaluation of filler by testing filler itself

Until 1932, only the percent passing number 200 sieve (- # 200) was used to evaluate and control mineral fillers used in asphaltic paving mixtures. Since 1932, it was proved that screens were useless in the analysis of filler because particles less than 40 μ were considered very important. The evaluation of fillers has included first the consideration of their primary properties, such as particle size, size distribution, particle shape and texture, and then the relation of these to the secondary properties of the filler, namely, surface area, packing of the particles and void content, and average void size in the compacted mineral powder.

Several tests which are applied to soils to show their physical characteristics were believed to be of considerable value in evaluating mineral fillers (12, 83, 88). These include standard tests such as hydrometer test, shrinkage ratio, liquid limit, plastic limit, and plastic index.

A number of different methods are available for measuring the primary properties of the filler (6, 24, 49, 68). However, hydrometer method has been used most widely, especially for routine samples, for determining the particle size because of its inexpensive equipment, simple operation and satisfactory results.

The shrinkage ratio is indicative of the void filling capacity of the filler.

The liquid limit, plastic limit, and plastic index together indicate the cohesive properties of a filler. A range of 25 to 50 has been proposed for the liquid limit of the filler material passing No. 200 sieve. The relative amount of cohesive of the filler is indicated by $(PI - \frac{LL}{4})$. (12, 88) All the three tests are described in A.A.S.H.O. and A.S.T.M. Standard Methods (2, 4).

Although it was believed that the function of filler in bituminous mixes was mechanical and that geometrical properties of fillers were more significant than chemical differences between various fillers (63, 74), the importance of chemical nature of the mineral fillers were stressed by some investigators (21, 79, 80). Since asphalts by nature are slightly acidic (32, 80), they show better adherence to alkalic than to acidic surfaces. Fillers with chemical affinity for asphalt such as calcareous fillers tend to disperse in asphalt, on the other hand, hydrophilic fillers, such as fine silica sand, have been found to have rather serious detrimental effects on the mix. (21, 83)

Ebberts (28) divided fillers into two classes: those which, when agitated with water and bitumen in equal proportion, form emulsion with bitumen in the continuous phase, and those which form emulsion with water in the continuous phase. According to him, limestone, magnesium carbonate, hydrated lime, magnesium hydrate and portland cement belonged to the first group, while silica, iron oxide and gypsum belonged to the second group. He suggested that if a mixture was laid using silica filler, and was porous, the absorbed water may disperse the bitumen, thus uncovering the filler, and cause rapid disintegration of the mixture.

Unfortunately, there is little information available concerning the specific effects of the chemical composition of fillers on the properties of mixes and the methods of evaluating them, except the water-asphalt preferential test which is sometimes used to reject hydrophilic materials proposed for use as filler (35, 83).

No doubt particle shapes have some effect on the manner in which the filler tends to agglomerate, the manner in which particles adhere to aggregate surfaces, and their tendency to be suspended in asphalt. All of these may have some effect on the properties of the mix (21, 80). The evaluation of particle shape in absolute terms is difficult. The difficulty is compounded due to the fact that, for a particular filler, the shape may vary for different size particles. Microscopic examination of a filler can give a fairly good idea of the shape of the particles. But as yet no information is available as to how does the particle shape actually affect a bituminous mix.

Chaiken, Halstead and Olsen (16) investigated the use of various instrumental methods such as DTA, X-ray difraction and infrared spectroscopy in identifying and estimating the components of minerals present in the filler. But no attempt was made to relate the quantitative amounts of the various minerals present to the performance of the proposed fillers.

Cementation test was proposed by Bollen (12) as another evaluation method for the cohesion property of filler. Cementation was the average number of blows at which a specially prepared briquette failed. He also proposed that the cementation of the material passing the No. 40 sieve should not be more than 100 when the quantity PI - $\frac{LL}{4}$ was greater than 5.

Extensive research on the effect of filler on bituminous mixtures has been done by Lee (44) and Rigden (62) of the Road Research Laboratory, Department of Scientific and Industrial Research, Great Britian. Standard procedures of testing and specification have been adopted by the British Standards Institution. They evaluate the fillers by two physical parameters: (a) the relative compaction of the filler (Cv), and (b) the effective size of pore of the compacted filler ($\overline{r}$), both measure for the degree of compaction of the filler in a filler-bitumen system. The effective size of pore of the compacted filler is related to the fineness of the filler: the finer the filler, the smaller is the pore size. A simple measure of the fineness of the filler was developed known as the bulk density in benzene in which the settled volume of 10 grams of filler is determined. Thus a high settled volume (low bulk density) shows the filler is fine. The upper limit of 0.95g/ml set on the bulk density of filler in B. S. 594 (14) is included to prevent too coarse a filler being used. The lower limit of 0.5g/ml is to exclude too fine a filler, which would give rise to coating difficulties and require an unduly high binder content.

Other tests included shrinkage limit, field moisture equivalent, sand equivalent test, cleanness test, capillarity, etc. Each has been used by some states in filler evaluation, but interpretation of results in terms of paving mix design has not been conclusive (83, 88).

Evaluation of filler by testing filler-asphalt mastic system

Many investigators (50, 80, 88) believed that the best way to obtain the information as to how a filler will affect an asphalt is to mix the asphalt and mineral powder and compare the properties of the filler-asphalt mixture with that of the asphalt.

Beginning in 1932, a series of papers by Traxler *et al.* (49, 50, 80, 81, 82, 83, 84, 85), presented a detailed study of filler, following the above reasoning. They measured the flow properties of filler-asphalt mixture in terms of absolute viscosity and found the viscosity of a viscous liquid--solid mixture was inversely proportional to the average void diameter of the filler as present in the mixture. Since size, size distribution and shape of the particles of the filler determined this average void diameter or void content, they were listed as the primary physical properties of fillers.

They also proposed to evaluate fillers by stability index, defined as the percent increase in viscosity resulting from an increase of one percent by volume of filler and was expressed as

$$S.I. = 100\ (10^{A} - 1)$$

where "A" was the slope of the volume percent of the filler vs. log viscosity plot. Their investigation also showed that the stability index of a filler was independent of the nature and viscosity of the asphalt present, provided no chemical reaction occured between the asphalt and dispersed filler.

In corelating the void diameter and stability index, methods of determining bulk density, bulk specific gravity and percent voids were also developed (82, 83).

Bollen (12) proportioned filler and bitumen by weight, and evaluated the stabilizing action of the filler, or the increase in viscosity effected by the addition of filler, by a float test. This test involved the determination of the filler-bitumen ratio by weight required to produce a float time of 1,000 seconds in a mixture of filler and SC oil under special temperature conditions. The "S" factor of the filler was defined by the relation

$$"S" = \frac{0.27}{\text{F.B. ratio}}$$

The stabilizing effect of filler was then used in the determination of the asphalt content of the mixture in such a manner that increased "S" factor resulted in higher asphalt contents.

Taylor (76) evaluated the filler in terms of the rise in softening point of filler-asphalt mastic and particle size of the filler. He found that the relative stabilizing powers of the fillers varied with the proportion of filler used. He showed that the rise in softening point caused by material ground to different degrees of fineness was not inversely proportional to the average diameter of the particles, but gave the relationship:

$$R = K/\sqrt{D}$$

where R = the rise of softening point of the asphalt in $^{\circ}$F.

D = average diameter of the filler particles

K = stabilizing power of the filler, varying with type of filler and the filler-asphalt ratio

The influence of fillers on the viscosity of bituminous binders was also studied by others. They have indicated that, in a general way, the properties of filler-binder system are largely governed by the bulking or packing properties of the filler. Mitchell and Lee (51) evaluated the filler in terms of viscosity of the filler-binder mastic. They found that, for fillers which were not soluble in bituminous binders, the addition of equal bulk volumes of different fillers to the same quantity of bituminous binder gave products having the same viscosity, irrespective of the nature of filler. The bulk density of a filler allowed to settle in a liquid is determined by the wetting properties of the liquid and the grading and geometrical properties of the filler particles. Mechanical tests on filler-binder mixes and full-scale road experiments have also shown that surfacings having similar properties could be obtained if one filler was substituted by another of the basis of the filler-binder combination being equal both in volume and viscosity.

In an attempt to find a simple characteristic of the filler which could be directly related to the behavior of filler-binder mixtures, Rigden (63) investigated the flow properties of a number of filler-binder mixtures over a range of filler concentration from zero to the greatest amount which gave a cohesive mixture. It was found that the relation between viscosity and filler concentration of any filler-binder system could be most simply interpreted if the concentration of the filler was expressed in terms of the degree of compaction of the filler as calculated from the fractional voids.

Fractional voids was the volume of voids, expressed as a decimal fraction, in a unit bulk volume of compacted filler.

Other than the flow property change due to the addition of filler, Evans (30) found the tensile strength of the filler-binder mastic was raised by the following factors:

(a.) Increase in the proportion of the filler.

(b.) Increase in the hardness of the bitumen.

(c.) Increase in the fineness of the filler.

(d.) Fibrous or lamellar as distinct from cubical structure of filler.

(e.) Increase in the bitumen adsorption power of the filler.

Ridgen and Lee (64) also found that the addition of mineral filler to an asphalt increased its brittle tensile strength. They concluded that the development of conditions for brittle fracture of the binder in a road surfacing were determined primarily by the effect of temperature, and of exposure to weather, and the viscosity of the binder.

In his study of dense bituminous road carpet design, Nijboer (55) found that satisfactory mixtures were characterized by "bitumen-filler mortars" possessing an optimum softening point of 181 °F. This implied an optimum ratio of bitumen to filler for any particular bitumen, and the ratio depended on the grading of filler but independent of mineralogy of the filler.

Strieter (75) compared the durabilities of filled and unfilled coating asphalts, both in outdoor and in accelerated exposures. The tests showed that in general the durability to weathering of coating asphalt could be improved by the addition of mineral fillers and that there was a difference in the effectiveness of various fillers.

Oliensis (57) reported his results on the effect of filler on asphalt durability in 1947. He had exposed coatings outdoors on asphalt-saturated felt for approximately ten years. These results indicated that fillers did stabilize the flow and thereby improve the life of roofing cements of relatively low softening point, which had a tendency to flow or sag when unfilled; but that fillers did not seem to improve the crack resistance of coatings of higher softening point which did not flow when unfilled.

Zapata and Hazmbeg (90) also studied the effect of filler on the durability of asphalt by exposing the mixture to natural weathering. They found that type and particle shape, rather than gradation, of a filler appeared to be more significant in the development of cracks in the films of filler-asphalt mixes. They also concluded that the addition of properly selected fillers could eliminate the bad cracking, reduce the porosity and improve the toughness of the film.

Evaluation of filler by testing asphalt mixtures

Survey made in 1937 (83) showed that twelve out of forty-eight states were using stability tests to evaluate mineral fillers, eight of which used Hubbard--Field method.

Carpenter (15) evaluated the fillers by measuring the unconfined compressive strength on asphaltic concrete specimens. He rated traprock dust and lime stone dust as good fillers, and fly ash superior to all the others. The principal test characteristic upon which the ratings of the fillers were based was the resistance of the compacted asphaltic concrete mixtures to loss of compressive strength after immersion in water at 120 ^{o}F for 4 days.

McLeod (48) evaluated fillers in terms of percent voids filled with bitumen, percent voids in the compacted mineral aggregate, unit weight, and Marshall stability and flow. The results of incremental increases in filler content up to 10 percent were shown to be beneficial in all respects, with little or no change at 15 percent, but the bitumen content must be reduced very substantially as percent filler was increased. He concluded that the mineral filler was a void-filling material, and that it could be highly detrimental to paving mixtures made with fine and coarse aggregates that in themselves resulted in very dense grading.

Warden *et al*. (88) evaluated the effect of filler on consistency, ductility, Marshall stability, resistance to water, and temperature susceptibility. Consistency and ductility were measured on filler-bitumen mortars, and the remaining characteristics were measured using a mixture containing Ottawa Sand. This investigation differed from others in that too great a change in consistency was considered to be detrimental.

Similar approach was adopted by Kallas et al. (38, 40) except that water susceptibility was measured on the basis of water absorption and reduction in stability of specimen soaked at 140 °F for 18 hours, and that the viscosity of filler-asphalt mixture was measured by sliding plate micro-viscometer. Also they used mechanical gyratory compactor and Hveem stability and cohesion value. Their conclusion was that type of mineral filler greatly influenced the compaction characteristics of paving mixture, the maximum Marshall stabilities as well as Marshall flow values, that pronounced differences in viscosity and shear susceptibility resulted when different fillers are mixed with asphalt; and that a general trend of increasing Marshall stability with increasing binder (asphalt and filler) viscosity resulted for both sheet asphalt and asphaltic concrete mixtures containing different fillers.

In an attempt to establish a mathematical relationship of filler (quantity times type) to the Marshall stability of an asphaltic concrete mixture using sand-gravel aggregate, Hudson and Vokac (37) introduced a coefficient K, defined as the ratio of the bulk volume of filler in benzene to the solid volume of the filler. The relationship were found as:

$$\text{Marshall Stability} = 660 + 250 \left(\frac{Vf}{Vb} - 0.25 \right) (K + 3.6)$$

where Vf/Vb is the ratio of filler to bitumen by volume.

Ekse (29) investigated the influence of varying amounts of filler on flexural strength and temperature susceptibility of compacted asphaltic concrete mixtures. He found that the flexural strength increased with increasing filler-asphalt ratio to a maximum when filler-asphalt ratio reached to about 1.5. His investigation also indicated a reduction in temperature susceptibility of the filler-asphalt mixture with increasing amounts of filler.

The effect of filler on stability and other properties of bituminous mixtures were also studied by other investigators (17, 34, 36, 37, 39, 77, 87).

Full scale test roads

In order to determine the significance of the conclusions resulting from their laboratory investigation dealing with the effect of fillers, the Road Research Laboratory of the Department of Scientific and Industrial Research, Great Britain, in co-operation with the British Tar Association (51), laid 164 bituminous surfacings containing both tar and asphaltic bitumen binders on the public road. Four commercial fillers had been employed, viz., limestone, slate, granite and portland cement. Comparative sections had been laid in which the same solid volume of the different fillers had been used, the quantities of the other constituents remaining the same.

Results from road test showed that the bulk density of a filler was its significant property in relation to bituminous road surfacing materials, and that failure may easily occur through neglect to adjust the filler-binder ratio on this basis. Surfacings having similar properties were obtained if one filler was substituted by another on the basis of the filler-binder combination being equal both in volume and viscosity.

Evaluation of filler by test road in the field was also carried out by the Corps of Engineers (87). They concluded that the material passing the number 200 sieve should be well-graded and that it includes 10 to 22 percent finer than 0.005 mm. It was reported that the proportion of filler had little effect on test track sections with high quality base materials, but that higher filler content appeared to be beneficial on sections with low quality bases.

Test track incorporated with asbestos as filler was laid in the Johns-Manville Mine in Quebec on a road used by ore trucks carrying ore from the mine to the mill (7). This pavement contained 2% asbestos fiber and was in good condition five years later when the control strip, without asbestos, had to be resurfaced because of extreme raveling and pitting.

Similar test roads for the purpose of studying asbestos used as filler in bituminous pavements were laid in the State of Delaware (27), the State of Georgia (56), and other places (31).

Types of Mineral Fillers

Materials which would perform satisfactorily as filler were offered by Richardson (60) as far back in 1905 including: portland cement, ground limestone, ground shale, and ground clay. Unsatisfactory or questionable materials were: ground waste lime from beet sugar factories, ground marl, ground silica, caustic or slated lime, and natural hydraulic cement. Ground clay was believed to offer the most potential as a filler because much of the natural filler in Trinidad asphalt was clay. Ground silica was listed as questionable because asphalt did not adhere to it as well and it could not be ground as fine. Later, acceptable filler materials were extended to include ground traprock, marl and volcanic ash (59).

Portland cement and limestone dust were also recommended as the best fillers by many other investigators (9, 13, 83, 87, 88) for various reasons.

The Committee on Present Practice of the Association of Asphalt Paving Technologists presented a report on fillers in current specifications in 1932 (9). This report revealed that a large number of materials were in use as fillers at that time, the most popular being limestone dust, portland cement, slate dust, stone dust, dolomite dust and silica dust, in addition to 17 others. The requirements specified for fillers varied, but all fillers were required to pass the No. 30 sieve with from at least 60 to 90 percent passing the No. 200 sieve.

A list of acceptable filler materials was offered by Spielman and Hughes (74) in 1936, which, headed by limestone dust and portland cement, included: slate dust, silica flour, brick dust, granite dust, flue dust, slag, asbestos, anhydrite, fuller's earth, barytes, coal dust, and coke dust, or anything fine enough to remain in suspension in the bitumen and chemically inert.

In 1937, Traxler, Bollen and Olmstead (83) presented an up-to-date survey of present practice, and the results of a cooperative testing program conducted in their respective laboratories. The survey disclosed that most of the states used basic fillers (cement, limestone, dolomite, etc.) in the higher types of construction. The fillers used in low-cost construction were predominantly basic although other local fillers were used in numerous cases. The merits of calcareous versus non-calcareous or acidic minerals were quite controversial, although the trend was in favor of the former type. The types of mineral fillers used by different states were, in order of popularity: limestone, cement, silica, dolomite, traprock, clay, volcanic ash, silt, loess, diatomaceous earth, stack dust, top soil, and nine others. The results of their cooperative testings, rejected diatomaceous earth, mica, green slate and clay, because of two or more of the following detrimental properties: (a) percentage finer than five microns was higher than 35; (b) percentage finer than one micron was higher than 15; (c) liquid limit was higher than 50; (d) "S" factor was higher than 0.35; (e) excessive swell in the swell test; (f) did not meet water-asphalt preferential test.

Materials employed as fillers given by Spielmann and Elford (73) were: brick dust, clay, coal dust, cyclone dust, diatomite, granite, limestone, portland cement, silica, slate magnesia, gypsum, anhydrite, iron oxide, strontium sulphate, barytes, etc.

Other materials that have been studied by various investigators for the purpose of filler in bituminous mixes include: fly ash (88), epure (30), ground chalk (30), ground silica (30, 37, 74, 79), stockalite (30), fuller's earth (40), Kaolin clay (37, 40, 82, 88), activated alumina (65), diatomaceous earth (38, 82), graphite (82), loess (12, 87), soapstone and talc (82), bentonite (88), and wood flour (82).

Kietzman (41) and many others (30, 31, 38, 71, 77, 79, 88) investigated the use of asbestos as a filler in bituminous paving mixtures. It appears that substantial benefits in the form of increased pavement life and performance and decreased maintenance costs can be achieved by the addition of 2 or 3 % by weight of a particular grade of asbestos. It has been claimed that such mixtures (a) give high Marshall and Smith triaxial stability and high tensile strength, (b) have higher resistance to plastic deformation, (c) give a tough surface with increased resistance to indentation at high temperatures under heavy loads, (d) are less brittle at low temperatures, (e) are more flexible and resilient, and (f) are more resistant to cracking.

As for the present practice, A.A.S.H.O. Standard Specifications for Mineral Fillers for Sheet Asphalt and Bituminous Concrete Pavements (2) requires that:

> The mineral filler shall consist of limestone dust, portland cement, or other inert mineral matter from sources approved by the Engineer.

The A.S.T.M. Tentative Specifications for Mineral Filler for Bituminous Paving Mixtures (5) requires that:

> The mineral filler shall consist of limestone dust, portland cement, or other suitable matter.

The Asphalt Institute Specifications for Mineral Filler (8) requires that:

> Mineral filler shall consist of finely ground particles of limestone, hydrated lime, portland cement, or other approved nonplastic mineral matter.

Materials used as mineral fillers are listed by the Highway Engineering Handbook (89) including, limestone dust, volcanic ash, silt, powdered shale, portland cement, mineral sludges, hydrated lime, rock flour, diatomaceous earth, and fly ash.

INVESTIGATION

Materials

Fillers

Fillers selected for this experiment were limestone dust which has been the most popular filler both in Iowa and other states, and loess which is most abundant in Iowa and could be a potential filler in Iowa if it proves to be satisfactory.

The loess used in this research project was obtained from a natural deposit located in Carroll County, Iowa. After being excavated from its source, the material was shipped to the laboratory with no attempt to control its physical state. When received, it was found to have about 8% moisture, and ranged in size from fine powder to lumps approximately 3 inches in diameter. The moisture content of Iowa loess in its natural state varies from 7 to 28%, and it is a mixture of fine quartz, grains of feldspar, and clay minerals of predominantly montmorillonite (26).

Prior to incorporating the raw loess into a test mixture, it was first converted into a dry mineral dust by drying and pulverizing operations. The drying step was accomplished by placing the moist loess in pans on top of a thermostatically controlled hot plate at 550 °F for 24 hours. After drying, the loess, which was still quite lumpy and hard, was then run through a laboratory hammer mill pulverizer to convert it to a satisfactory fine powder. The material, as discharged from the hammer mill, was found to be 46 % passing No. 200 sieve, and had a moisture content of less than 1 %. The commercial feasibility of producing loess as a filler was tested both in the laboratory and in the field, and it was found that loess could be easily and economically processed into a suitable mineral dust (20, 22).

Limestone dust was obtained from a continuous hot mix plant located near Ocheydan, Iowa, where it was actively engaged in producing material for Iowa Route Number 9 in accordance with Iowa State Highway Commission Contract Number FN-329.

The X-ray diffraction test indicated that the predominate mineral components were dolomite, quartz and calcite.

Since all the secondary properties, which may effect the filler-asphalt system greatly, are due to the fineness of the filler particles, and since at present time the definition of filler is not very well clarified and agreed upon by asphalt paving technologists, two filler sizes, passing number 200 sieve (74μ) and passing number 325 sieve (44μ), are used for each kind of filler. Both fillers were screened in the laboratory and oven dried at 220 °F for 24 hours and cooled in desicator before use.

The physical properties of Carroll County loess and Ocheydan limestone dust are presented in Table 1. Specific

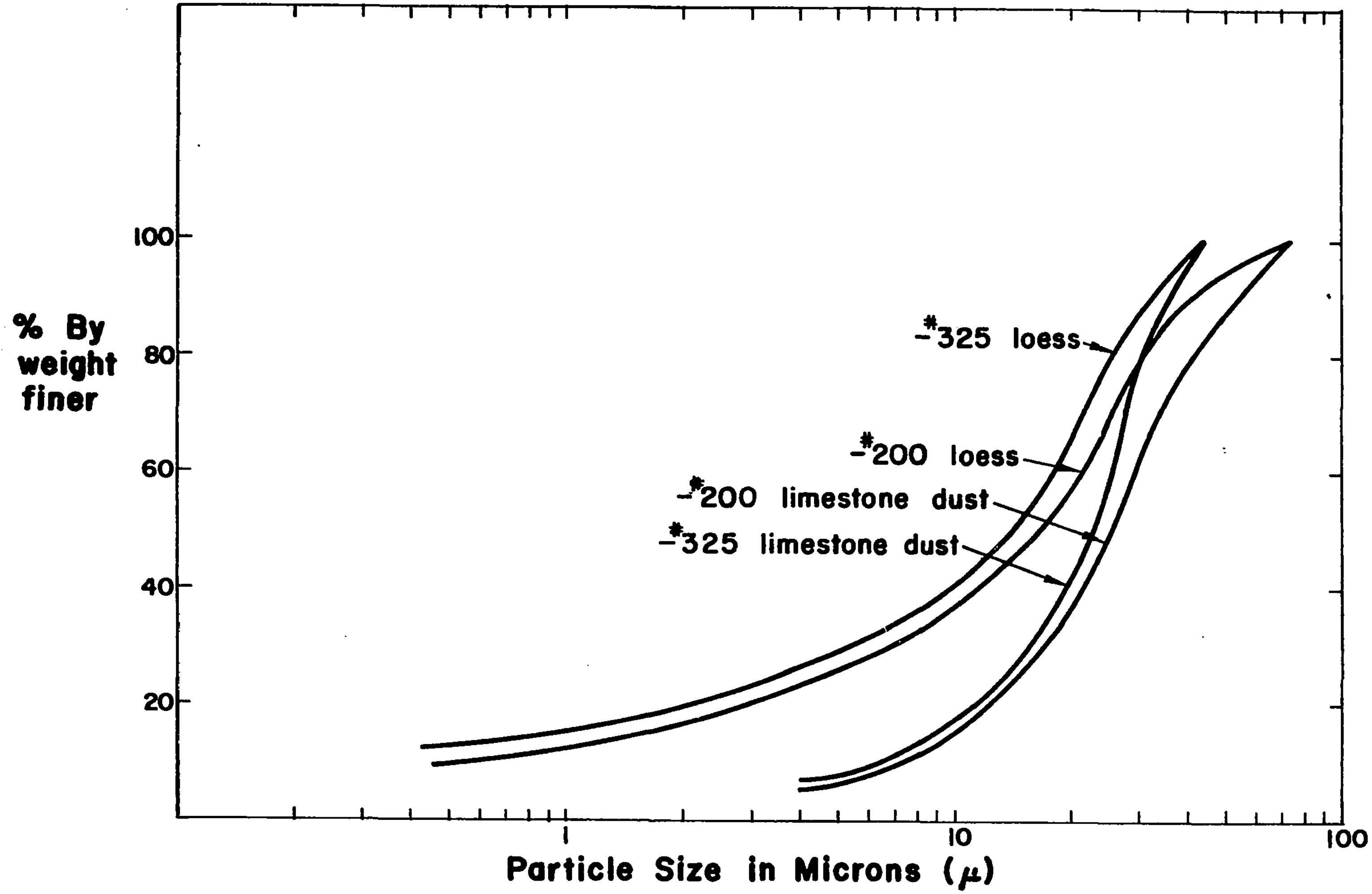
Table 1. Properties of fillers

Type	Grade	Source	Specific gravity	Voids %	Bulk density[a] g/ml
Limestone dust	-#200 sieve	Ocheyden, Iowa	2.68	46.4	0.96
	-#325 sieve		2.76	49.3	0.88
Pulverized loess	-#200 sieve	Carrol Co., Iowa	2.63	52.5	0.83
	-#325 sieve		2.60	54.2	0.78

[a] Mitchell and Lee (51).

gravity was determined by A.S.T.M. C 188 using the Le Chatelier flask and kerosene as the liquid. The differences of specific gravity between the passing number 200 sieve filler and that of the passing number 325 sieve filler are probably due to the differences in mineral composition between the two size portions. The procedure for determining air voids in the filler is given in the Appendix (88). The particle size analyses of the fillers were determined by hydrometer method, using an air-jet stirrer and with sodium metaphosphate as the dispersing agent, in accordance with A.S.T.M. D 422-54T. The gradation curves were plotted in Fig. 1. Bulk density was included because it has been indicated by many investigators (44, 51, 62) that, in a general way, the influence of any filler on the

Fig. 1. Particle-size distribution of mineral fillers


% By weight finer
100
80
60
40
20
1
10
100
Particle Size in Microns (μ)
-#325 loess
-#200 loess
-#200 limestone dust
-#325 limestone dust

properties of asphalt is largely governed by its bulking or packing properties. Bulk density was determined by method proposed by Mitchell and Lee (51). The procedure is also described in detail in the Appendix.

Asphalt cements

The asphalt cements used for this work were all straight-run asphalt cements having the properties shown in Table 2.

Test Procedure

The experimental work consisted of testing the effect of four fillers on the properties of two penetration grades asphalt cements. The fillers were added to the asphalt in proportions of 1, 2 1/2, 5, 10, 15, 20, and 40 % by weight of asphalt cement.

The eight combinations of filler and asphalt were:

85 - 100 pen. A.C. + -# 200 limestone dust
85 - 100 pen. A.C. + -# 325 limestone dust
85 - 100 pen. A.C. + -# 200 loess
85 - 100 pen. A.C. + -# 325 loess
120 - 150 pen. A.C. + -# 200 limestone dust
120 - 150 pen. A.C. + -# 325 limestone dust
120 - 150 pen. A.C. + -# 200 loess
120 - 150 pen. A.C. + -# 325 loess

Table 2. Properties of asphalt cements

Properties	ASTM test method	85-100 pen.	120-150 pen.
Specific gravity at 77 °F	ASTM D-70	1.020	1.012
Penetration 77/100/5	ASTM D-5	88	126
Flash point °F	ASTM D-92	637	615
Softening point, °F	ASTM D-36	120.2	111.2
Ductility 77 °F, cm.	ASTM D-113	150+	150+
Solubility in CCl_4, %	ASTM D-165	99.86	99.75
Penetration index[a]		-0.1	-0.5
Asphaltene content[b]		21.2	18.7
Viscosity[c] centipoise			
at 225 °F		1,400	900
at 275 °F		320	200
at 325 °F		130	70

[a] Pfeiffer and van Doormaal (58).

[b] Csanyi and Fung (23).

[c] Brookfield viscometer.

Preparation of mixtures

Filler-asphalt mastic samples were prepared in the laboratory by heating about 800 grams of asphalt cement in a beaker to 325 °F then adding the filler, preheated to 325 °F, in quantities of 1, 2 1/2, 5, 10, 15, 20 and 40 % by weight of asphalt cement.

The filler was added in a slow thin stream and stirred in by means of a mechanical stirrer rotating at a speed of about 400 rpm. The filler was introduced into the bitumen from about 1" above the surface of the bitumen so that the finest part of the filler would not be lost into the air. When all the filler had been incorporated, the mixture was stirred for a further 15 minutes, then followed by stirring with a spatula for 1 minute.

It was found, by a series of trials, that the mixing will be complete in 15 minutes, and the additional stirring by spatula will help distribute the filler more uniformly at the bottom and sides of the container. The temperature was kept between 300 °F and 325 °F throughout the course of mixing. The mixture was then poured into an aluminum cylinder 2 1/2 inches in diameter and 11 inches tall. The mixture was then placed in oven at 325 °F for 24 hours, cooled to room temperature and put in freezer at -20 °F for 24 hours. After that, the frozen mixture was divided into three equal layers by a tube cutter, and coded as T, M and B representing top, middle and bottom layer respectively. Finally, the mixture in each layer was transferred into a beaker by gentle heating in preparation for testing.

Methods of testing

The following tests were run on each and every layer. Selection of test procedures and equipments was based on the simplicity of operation and control.

Penetration	77°F/100grms/5 sec	A.S.T.M. D 5
Softening Point	Ring and ball	A.S.T.M. D 36
Ductility, cm	77°F, 5 cm/min.	A.S.T.M. D 113
Extraction, (filler content)		Soxhlet extractors
Viscosity	325 °F,	Brookfield viscometer
	275 °F	
	225 °F	

For penetration, softening point, ductility and extraction tests, the mixture was gently reheated with constant slow stirring by spatula to redisperse settled filler and to get it just fluid enough for transfer into penetration containers, the softening point rings, the ductility molds and extraction thimbles. The consistency of the mixture and the manner of stirring were so adjusted to minimize the formation of air bubbles and at the same time to keep the filler from settling out.

The purpose of extraction tests was to determine the dust content in each layer permitting calculation of settlement percentage and estimate of amount of suspension.

Soxhlet extractors and Whatman extraction thimbles of double thickness were used for all the extractions. Samples from 20 to 80 grams were used depending upon layers and filler contents. Benzene was selected as the solvent because of its light specific gravity compared with carbon tetrachloride. Each extraction was completed in about 36 hours.

Viscosity was determined by Brookfield viscometer (Model LV) which has six spindles of different sizes and four rotating speeds (2, 4, 10 and 20 rpm). This equipment measures the drag produced upon a spindle rotating at a definite constant speed while immersed in the sample under test. For each sample, viscosity readings were taken from 350 ^{o}F down to 200 ^{o}F, with mixture stirred and redispersed at before 325 ^{o}F, 275 ^{o}F and 225 ^{o}F. To prevent over heating of the sample resulting possible asphaltene content change in the asphalt, the samples were heated to temperatures well below 400 ^{o}F. Because of limited quantity of sample in each layer, the specimens used for ductility tests had to be reused for viscosity tests.

Penetration index after Pfeiffer and Doormaal (58) was also determined.

RESULTS

Two most commonly used asphalt cements, 85-100 pen. and 120-150 pen., two type of fillers, the most popular filler limestone dust and a locally available filler loess were used in this study. In addition, two particle sizes, of fillers, -# 200 sieve (74μ) and -# 325 sieve (44μ), were also included. Eight mastics with varying filler contents, for each and every one of the eight filler-asphalt combinations were prepared. Each of the three layers of every mastic mixture was subjected to penetration, ductility, softening point, and viscosity test. Hot extractions were run for each layer in order to determine the filler contents in suspension.

Test results have been summarized and tabulated in Table 3 through Table 10. Curves have been plotted in Fig. 2 through Fig. 14. The amount of filler in each layer determined by extraction was expressed in percentage by weight of asphalt and was used in plotting curves involving filler concentrations.

For the penetration data, the average of three tests of any one specimen was used. If one of the three values obtained differed more than 2 units from the average, this was discarded in the calculating of the average. Ductility data were the average of results of duplicate tests, check tests were run for tests that differed more than 10% of the average. The softening point data represents also the average of two tests. Check tests were made if the results of the duplicate tests differed more than 1/2 °C from the average.

The viscosity at 325 °F, 275 °F and 225 °F for each specimen were taken from corrected log viscosity versus log temperature curves for this specimen over the range of 200 °F through 350 °F.

The amount of filler remaining in top and middle layers after 24 hours settlement at 325 °F divided by the total amount of filler in that mix expressed in percentage was taken as the percent in suspension, and the term colloidal suspension was so defined.

Table 3. Properties of mastics containing 120-150 pen. A.C. and -#200 limestone dust

Sample no.	% Filler added	% Filler by extr.	Penetration 77/100/5	Softening point, °F	P.I.	Ductility cm.	Viscosity, poise		
							225°F	275°F	325°F
S211	0.00	0.00	126.0	111.2	-0.5	150+	9.0	2.1	0.7
S212 T	1.00	0.17	124.0	111.2	-0.5	150+	9.0	2.0	0.7
M		0.21	125.5	112.1	-0.3	150+	9.0	2.1	0.7
B		2.41	125.0	111.2	-0.5	120.0	9.4	2.1	0.7
S213 T	2.50	0.38	128.0	111.2	-0.5	150+	9.0	2.1	0.7
M		0.23	127.0	110.3	-0.6	150+	9.0	2.1	0.7
B		3.47	121.0	111.2	-0.6	115.0	9.5	2.2	0.8
S214 T	5.00	0.53	127.0	111.2	-0.5	150+	9.3	2.1	0.7
M		0.46	126.0	111.2	-0.5	133.0	9.2	2.1	0.7
B		14.56	120.0	113.0	-0.4	74.0	11.0	2.6	0.8
S215 T	10.00	1.15	125.5	112.1	-0.3	150+	9.2	2.2	0.8
M		1.36	125.0	112.1	-0.3	135.0	9.2	2.2	0.8
B		25.68	115.0	114.8	-0.2	64.0	12.7	3.2	1.1
S216 T	15.00	1.67	124.0	111.2	-0.5	146.0	9.1	2.1	0.8
M		2.02	124.0	112.1	-0.5	150+	9.3	2.2	0.8
B		34.40	110.5	115.7	-0.1	60.0	14.4	3.7	1.2
S217 T	20.00	1.86	124.0	112.1	-0.5	146.0	9.2	2.1	0.7
M		3.09	123.0	113.0	-0.2	115.0	9.5	2.1	0.8
B		52.17	103.5	118.4	+0.2	52.0	19.0	5.5	1.8
S218 T	50.00	4.04	123.0	113.0	-0.4	115.0	9.3	2.2	0.8
M		10.47	121.0	114.8	+0.1	83.0	10.6	2.4	0.9
B		197.52	43.0	177.6	+4.1	2.0	1,240.0	240.0	180.0

Table 4. Properties of mastics containing 120-150 pen. A.C. and -#325 limestone dust

Sample no.		% Filler added	% Filler by extr.	Penetration 77/100/5	Softening point, °F	P.I.	Ductility cm.	Viscosity, poise		
								225°F	275°F	325°F
S311		0.00	0.00	126.0	111.2	-0.5	150+	9.0	2.1	0.7
S312	T	1.00	0.53	127.0	111.2	-0.5	150+	9.0	2.1	0.7
	M		0.43	126.0	111.2	-0.5	133.0	9.0	2.1	0.7
	B		2.71	123.0	112.1	-0.5	125.0	9.4	2.2	0.7
S313	T	2.50	0.41	127.0	111.2	-0.5	140.0	9.0	2.0	0.7
	M		0.77	126.0	111.2	-0.5	128.0	9.1	2.1	0.7
	B		5.62	122.0	112.1	-0.6	104.0	9.8	2.3	0.8
S314	T	5.00	1.06	126.0	111.2	-0.5	135.0	9.0	2.1	0.7
	M		1.13	125.0	112.1	-0.4	133.0	9.1	2.1	0.8
	B		9.91	120.0	112.1	-0.6	88.0	10.5	2.4	0.9
S315	T	10.00	1.66	125.0	111.2	-0.5	142.0	9.1	2.1	0.8
	M		2.97	124.0	112.1	-0.5	120.0	9.4	2.2	0.7
	B		24.40	114.0	113.9	-0.4	69.0	12.5	3.4	1.1
S316	T	15.00	2.12	124.0	111.2	-0.6	130.0	9.1	2.2	0.7
	M		3.88	123.0	112.1	-0.5	110.0	9.5	2.3	0.8
	B		42.25	106.0	115.7	-0.2	60.0	18.0	4.6	1.6
S317	T	20.00	2.41	126.0	112.1	-0.5	125.0	9.2	2.3	0.9
	M		4.01	123.0	112.1	-0.5	108.0	9.6	2.3	0.8
	B		65.82	95.0	119.3	+0.1	52.0	29.0	9.4	3.1
S318	T	50.00	5.39	123.0	112.1	-0.5	104.0	9.8	2.3	1.0
	M		25.42	116.0	113.9	-0.4	69.0	14.0	3.6	1.2
	B		145.89	45.0	146.0	+1.4	18.0	290.0	75.0	24.0

Table 5. Properties of mastics containing 120-150 pen. A.C. and -#200 loess

Sample no.	% Filler added	% Filler by extr.	Penetration 77/100/5	Softening point,°F	P.I.	Ductility cm.	Viscosity, poise		
							225°F	275°F	325°F
L211	0.00	0.00	126.0	111.2	-0.5	150+	9.0	2.1	0.7
L212 T	1.00	0.15	126.0	113.0	-0.2	142.0	9.0	2.1	0.7
M		0.21	126.5	111.2	-0.5	138.0	8.9	2.0	0.8
B		2.54	124.0	112.1	-0.6	90.0	9.5	2.2	0.8
L213 T	2.50	0.31	127.0	110.3	-0.7	150+	9.1	2.1	0.8
M		0.28	126.0	111.2	-0.6	140.5	9.0	2.1	0.8
B		6.47	121.5	112.1	-0.5	67.5	10.5	2.4	0.9
L214 T	5.00	0.34	125.5	111.2	-0.6	112.5	8.9	2.1	0.8
M		0.47	126.0	110.3	-0.7	126.0	9.0	2.2	0.7
B		11.51	118.0	113.0	-0.4	58.0	11.2	2.7	0.9
L215 T	10.00	0.82	126.0	111.2	-0.8	115.0	9.0	2.1	0.8
M		0.98	125.0	110.3	-0.7	120.5	9.1	2.2	0.9
B		20.91	110.0	114.8	-0.2	50.5	13.5	3.3	1.3
L216 T	15.00	1.54	126.5	112.1	-0.5	117.5	9.2	2.2	0.9
M		1.43	126.0	111.2	-0.5	127.5	9.3	2.2	0.8
B		30.94	102.0	121.1	-0.5	41.0	18.0	4.5	2.1
L217 T	20.00	1.94	124.0	111.2	-0.5	109.0	9.3	2.3	1.0
M		1.89	124.5	111.2	-0.5	102.5	9.3	2.3	1.0
B		38.97	91.5	124.7	+0.8	32.0	23.5	6.4	3.4
L218 T	40.00	3.88	123.0	112.1	-0.5	139.5	10.1	2.4	1.1
M		5.01	122.5	113.0	-0.3	92.0	10.3	2.5	1.2
B		58.94	47.0	161.6	+3.0	4.5	400.0	74.0	35.0

Table 6. Properties of mastics containing 120-150 pen. A.C. and -#325 loess

Sample no.		% Filler added	% Filler by extr.	Penetration 77/100/5	Softening point, °F	P.I.	Ductility cm.	Viscosity, poise		
								225°F	275°F	325°F
L311		0.00	0.00	126.0	111.2	-0.5	150+	9.0	2.1	0.7
L312	T	1.00	0.31	124.0	111.2	-0.5	140.0	9.0	2.1	0.7
	M		0.29	125.0	113.0	-0.5	138.0	9.0	2.0	0.7
	B		3.52	122.5	111.2	-0.6	115.0	9.2	2.2	0.7
L313	T	2.50	0.34	122.5	111.2	-0.6	136.0	9.0	2.1	0.7
	M		0.32	120.0	112.1	-0.6	126.0	9.0	2.1	0.7
	B		7.02	119.5	112.1	-0.7	92.0	9.7	2.3	0.8
L314	T	5.00	0.80	126.5	110.3	-0.6	128.0	9.0	2.1	0.7
	M		0.84	126.0	112.1	-0.5	145.0	9.1	2.1	0.7
	B		13.03	114.5	113.0	-0.6	74.0	10.0	2.5	0.9
L315	T	10.00	1.29	125.5	112.1	-0.5	139.0	9.0	2.1	0.7
	M		1.39	125.0	113.0	-0.5	130.0	9.0	2.1	0.7
	B		23.00	106.0	114.8	-0.3	57.0	11.9	3.0	1.2
L316	T	15.00	2.09	123.7	112.1	-0.5	144.0	9.1	2.2	0.8
	M		1.88	124.0	112.1	-0.5	150+	9.0	2.1	0.7
	B		32.11	98.0	117.5	-0.2	51.0	16.0	3.9	1.8
L317	T	20.00	2.96	125.4	111.2	-0.6	125.5	9.1	2.1	0.7
	M		3.92	125.0	111.2	-0.6	130.0	9.3	2.2	0.8
	B		36.87	91.0	121.1	+0.1	44.0	20.0	5.0	2.6
L318	T	40.00	3.92	123.0	111.2	-0.6	99.5	9.1	2.3	0.7
	M		9.05	108.0	113.0	-0.7	78.5	9.6	2.5	0.9
	B		56.13	50.0	143.6	+1.5	16.5	342.5	79.0	39.0

Table 7. Properties of mastics containing 85-100 pen. A.C. and -#200 limestone dust

Sample no.	% Filler added	% Filler by extr.	Penetration 77/100/5	Softening point, °F	P.I.	Ductility cm.	Viscosity, poise		
							225°F	275°F	325°F
S221	0.00	0.00	88.0	120.2	-0.1	150+	14.0	3.2	1.2
S222 T	1.00	0.12	87.0	120.2	-0.1	150+	14.0	3.1	1.1
M		0.15	87.0	120.2	-0.1	150+	14.0	3.2	1.2
B		2.68	85.0	120.2	-0.1	84.0	14.6	3.3	1.2
S223 T	2.50	0.27	87.0	120.2	-0.1	150+	14.0	3.1	1.1
M		0.26	88.0	120.2	-0.1	145.0	14.0	3.2	1.2
B		5.12	83.0	121.1	-0.3	71.0	14.0	3.2	1.2
S224 T	5.00	0.43	86.0	120.2	-0.1	149.0	14.0	3.2	1.2
M		0.47	85.0	120.2	-0.1	150+	14.0	3.2	1.2
B		11.21	77.0	122.0	-0.4	54.0	14.5	3.4	1.3
S225 T	10.00	0.94	86.0	120.2	-0.1	150+	14.0	3.2	1.2
M		0.94	84.0	120.2	-0.2	150+	14.0	3.2	1.2
B		21.56	71.0	122.0	-0.5	40.0	16.0	3.9	1.7
S226 T	15.00	1.20	81.0	120.2	-0.1	150+	14.0	3.2	1.2
M		1.19	79.0	120.2	-0.3	143.0	14.0	3.2	1.2
B		31.59	64.0	123.8	-0.4	29.0	18.9	5.2	2.4
S227 T	20.00	1.66	85.0	120.2	-0.1	147.0	14.0	3.2	1.2
M		1.89	84.5	120.2	-0.1	141.0	14.0	3.2	1.2
B		41.03	59.0	128.3	0.0	19.0	30.0	9.0	4.2
S228 T	40.00	2.38	85.0	121.1	-0.2	130.0	14.1	3.3	1.2
M		4.68	83.5	122.0	-0.2	117.0	14.2	3.3	1.2
B		62.23	33.0	159.8	+2.0	4.0	400.0	76.0	28.0

Table 8. Properties of mastics containing 85-100 pen. A.C. and -#325 limestone dust

Sample no.	% Filler added	% Filler by extr.	Penetration 77/100/5	Softening point, °F	P.I.	Ductility cm.	Viscosity, poise		
							225°F	275°F	325°F
S321	0.00	0.00	88.0	120.2	-0.1	150+	14.0	3.2	1.2
S322 T	1.00	0.25	87.0	120.2	-0.1	150+	14.0	3.1	1.1
M		0.34	87.0	120.2	-0.1	134.0	14.1	3.2	1.2
B		2.21	86.0	120.2	-0.1	90.0	14.5	3.2	1.2
S323 T	2.50	0.51	87.5	120.2	-0.1	138.0	14.0	3.2	1.2
M		0.51	87.0	120.2	-0.1	139.0	14.0	3.2	1.2
B		5.04	83.5	121.1	-0.1	73.0	15.0	3.4	1.3
S324 T	5.00	1.11	86.5	120.2	-0.1	150+	14.1	3.2	1.2
M		0.90	86.0	120.2	-0.1	134.0	14.0	3.1	1.1
B		9.65	80.5	121.1	-0.2	67.0	16.0	3.6	1.3
S325 T	10.00	1.63	85.0	121.1	0.0	150+	14.2	3.2	1.2
M		1.48	85.5	120.2	-0.1	133.0	14.3	3.2	1.2
B		19.81	75.0	122.0	-0.3	57.0	19.0	4.5	1.5
S326 T	15.00	2.01	85.5	120.2	-0.1	130.0	14.4	3.1	1.1
M		2.94	85.0	121.1	0.0	125.0	14.7	3.1	1.1
B		28.72	69.0	122.9	-0.4	50.0	24.5	6.0	1.9
S327 T	20.00	2.48	84.0	120.2	-0.2	129.0	14.6	3.4	1.3
M		4.51	85.0	121.1	0.0	108.0	14.6	3.4	1.4
B		35.94	62.5	125.6	-0.2	39.0	33.1	8.4	2.8
S328 T	40.00	3.56	84.0	120.2	-0.1	100.0	14.8	3.3	1.3
M		11.18	80.0	121.1	-0.2	81.0	16.8	3.9	1.4
B		52.22	45.5	136.4	+0.4	18.0	153.0	41.0	13.0

Table 9. Properties of mastics containing 85-100 pen. A.C. and -#200 loess

Sample no.	% Filler added	% Filler by extr.	Penetration 77/100/5	Softening point, °F	P.I.	Ductility cm.	Viscosity, poise		
							225°F	275°F	325°F
L221	0.00	0.00	88.0	120.2	-0.1	150+	14.0	3.2	1.2
L222 T	1.00	0.14	88.0	120.2	-0.1	140.0	14.0	3.1	1.2
M		0.57	88.0	120.2	-0.1	125.0	14.0	3.2	1.2
B		1.31	85.0	121.1	-0.1	106.0	14.5	3.2	1.2
L223 T	2.50	0.35	87.0	120.2	-0.2	150+	14.0	3.2	1.2
M		0.40	86.5	120.2	-0.2	150+	14.0	3.2	1.2
B		5.54	81.0	122.1	-0.1	63.0	15.5	3.4	1.3
L224 T	5.00	0.55	86.0	121.1	0.0	150+	14.0	3.2	1.2
M		0.56	86.0	121.1	0.0	130.0	14.0	3.2	1.2
B		13.39	77.0	122.0	-0.2	46.0	18.0	3.9	1.4
L225 T	10.00	1.51	85.0	121.1	0.0	96.0	14.7	3.3	1.3
M		1.34	85.5	121.1	0.0	100.0	14.6	3.2	1.2
B		22.28	69.0	123.8	-0.2	36.5	22.0	4.8	1.7
L226 T	15.00	2.24	84.0	121.1	0.0	88.0	14.8	3.3	1.2
M		2.00	84.5	120.2	0.0	90.0	14.6	3.2	1.2
B		28.87	64.0	127.4	+0.1	32.0	27.5	6.3	2.0
L227 T	20.00	2.84	83.0	121.1	0.0	78.0	14.5	3.3	1.2
M		3.15	82.5	122.0	0.0	76.0	14.5	3.4	1.3
B		38.60	56.0	134.6	+0.7	24.0	49.0	10.6	3.3
L228 T	40.00	3.99	82.0	121.1	-0.1	72.0	15.3	3.4	1.3
M		9.41	77.0	122.9	-0.1	54.0	16.3	3.7	1.4
B		59.30	30.0	181.4	+3.4	3.0	600.0	150.0	53.0

Table 10. Properties of mastics containing 85-100 pen. A.C. and -#325 loess

Sample no.		% Filler added	% Filler by extr.	Penetration 77/100/5	Softening point,°F	P.I.	Ductility cm.	Viscosity, poise		
								225°F	275°F	325°F
L321		0.00	0.00	88.0	120.2	-0.1	150+	14.0	3.2	1.2
L322	T	1.00	0.15	88.0	120.2	-0.1	150+	14.0	3.2	1.1
	M		0.48	87.5	120.2	-0.1	138.0	14.0	3.1	1.2
	B		1.88	83.0	121.1	-0.1	96.0	14.0	3.2	1.2
L323	T	2.50	0.45	86.5	119.3	-0.2	139.0	14.0	3.2	1.2
	M		0.40	86.0	120.2	-0.1	138.0	14.0	3.2	1.2
	B		6.24	79.0	121.1	-0.2	71.0	14.2	3.3	1.2
L324	T	5.00	0.85	85.0	120.2	-0.1	150+	14.0	3.2	1.2
	M		0.84	84.0	120.2	-0.1	141.0	14.0	3.2	1.2
	B		12.59	75.0	122.9	-0.2	54.0	15.0	3.6	1.3
L325	T	10.00	1.95	82.5	122.0	0.0	146.0	14.1	3.2	1.2
	M		1.81	83.0	121.1	-0.1	150+	14.0	3.2	1.2
	B		24.28	66.0	124.7	-0.2	49.0	17.0	4.5	1.5
L326	T	15.00	2.35	81.5	121.1	-0.1	144.0	14.0	3.2	1.2
	M		3.78	80.0	122.0	0.0	126.0	14.1	3.2	1.2
	B		32.12	59.0	127.4	-0.1	44.0	22.0	5.6	1.9
L327	T	20.00	3.08	80.5	122.0	0.0	129.0	14.1	3.1	1.2
	M		4.16	79.0	121.1	-0.2	123.0	14.1	3.2	1.2
	B		39.26	49.0	132.8	+0.2	36.0	33.0	7.7	2.7
L328	T	40.00	4.54	79.0	121.1	-0.2	125.0	14.2	3.3	1.2
	M		12.72	73.0	122.0	-0.3	84.0	14.9	3.7	1.3
	B		53.15	30.0	151.7	+1.0	19.0	220.0	62.0	20.0

DISCUSSION

Penetration vs. Filler Content

Fig. 2 shows the relationship between penetration 77/100/5 of the mastics and percent filler by weight.

A general statement can be made from the curves that, very roughly, the penetration is inversely proportional to the percentage of filler present, or that the consistency is proportional to filler content of the mastic.

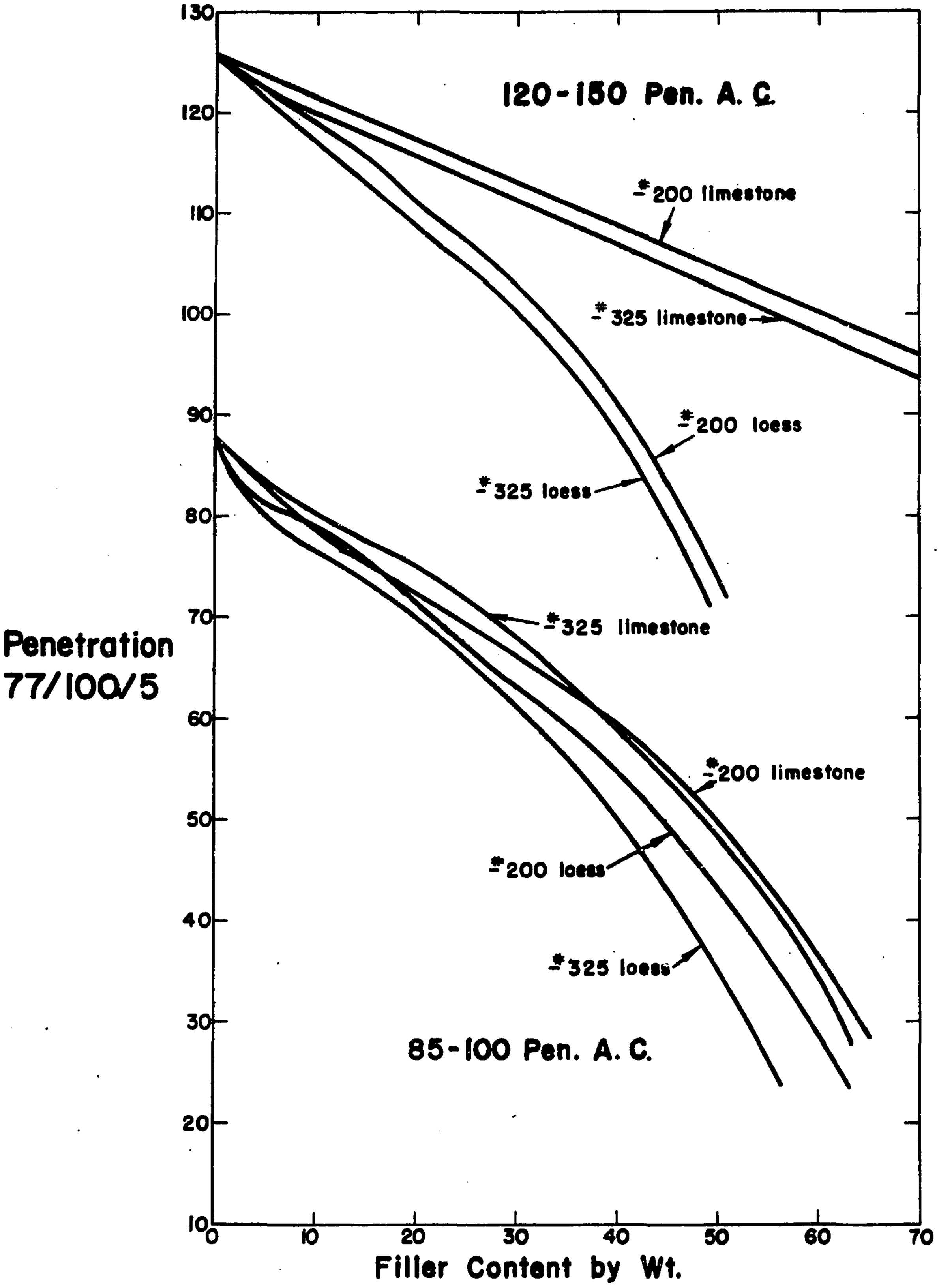
For 120-150 pen. A.C.

It is readily seen that two grades of loess fillers are more effective in decreasing penetration than either grade of limestone dust. This is especially true at higher filler content.

Over the practical working range studied the curves for the limestone approximate a straight line with the coarser grade filler (-# 200) having a lesser effect. The slope of these lines can be thought as a measure of the rate of change of penetration per unit weight of added filler. For limestone dust in 120-150 pen. A.C. the rate is about 0.4 units of penetration per 1% of filler added.

In the case of loess, straight line relationship holds only up to 30% filler, with a rate of about 0.8 units of penetration per 1% of filler added. After 30%, penetration

Fig. 2. Relationship between penetration of the mastics and percent filler by weight


120-150 Pen. A. C.
-#200 limestone
-#325 limestone
-#200 loess
-#325 loess
-#325 limestone
-#200 limestone
-#200 loess
-#325 loess
85-100 Pen. A. C.
Penetration 77/100/5
Filler Content by Wt.
10
20
30
40
50
60
70
80
90
100
110
120
130
0
10
20
30
40
50
60
70

decreases very rapidly and continuously to 60%, the maximum filler content studied for this combination. If this trend is extrapolated, a zero penetration would be expected at a filler content of about 70% to 80% by weight.

For 85-100 pen. A.C.

Though loess fillers are still more effective in decreasing penetration the change in consistency of asphalt cement has different effects on limestone dust than on loess. The slope or the rate of penetration change due to addition of 1% limestone dust is greatly increased, whereas the rate of penetration change due to addition of 1% loess is slightly decreased.

Regardless the consistency of the asphalt, the finer grade fillers (-# 325) are more effective in hardening or reducing penetration of the mastic. This can be explained by the larger surface area of the finer grade fillers which provides larger capacity for adsorption for bitumen, and because of the surface energy of attraction between the two materials, the adsorbed layer of bitumen assumes stiffer consistency. And it goes without saying that highest consistency will result as the total surface area of filler increases until all bitumen is influenced by adsorption without repulsion between coated filler particles.

The greater relative effectiveness of the loess fillers in reducing penetration of the mastic in comparison with that of the limestone fillers can also be partly attributed to the greater surface area of the former.

Though the bulk density of the filler and penetration of the mastics was not very well related, it can readily be seen that the relative effectiveness of the filler in reducing the penetration of the mastics does increase with the decreasing bulk density of the filler.

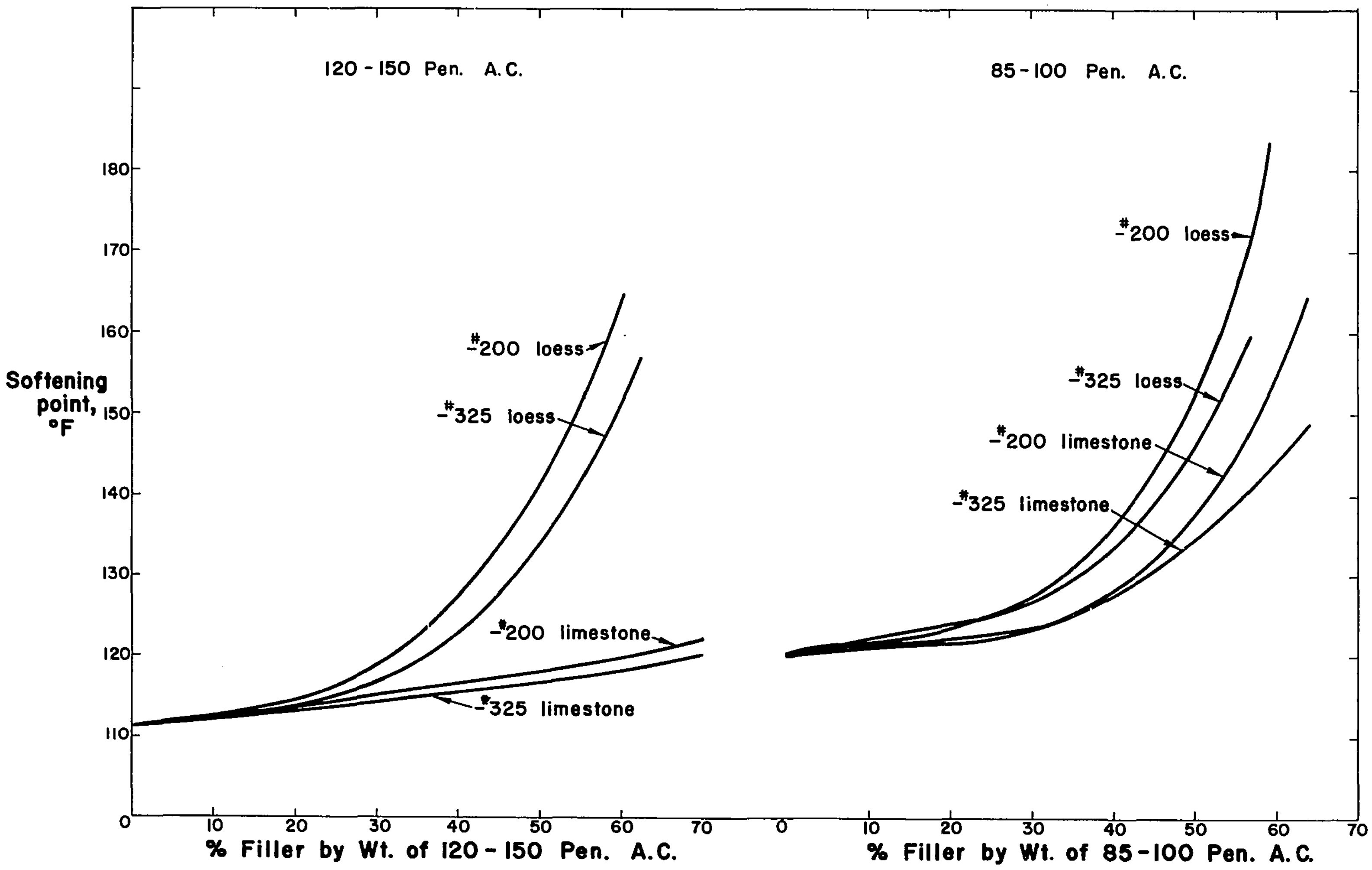
It is to be noted that there are irregularities, especially at low filler content. These are probably due to the limits of sensitiveness of the test. The results would also be expected to vary, to a certain degree, according to whether the needle came into contact with a filler particle or not while passing through the mixture.

It was also suspected that, during the preparation of the penetration sample, settling out of the filler might occur to an extent that would cause inconsistency of the results. In order to test for this, penetration tests were run both on the top and the bottom of a sample. The results showed that the variations were within the accuracy of the test and that very little settling had taken place. This was also confirmed by the Stoke's law.

Softening Point vs. Filler Content

Fig. 3 shows softening point vs. filler content by weight of 120-150 pen. A.C. and of 85-100 pen. A.C.

Fig. 3. Relationship between softening point of the mastics and percent filler by weight


120 - 150 Pen. A.C.
85 - 100 Pen. A.C.
Softening point, °F
180
170
160
150
140
130
120
110
0
-#200 loess
-#325 loess
-#200 limestone
-#325 limestone
-#200 loess
-#325 loess
-#200 limestone
-#325 limestone
10
20
30
40
50
60
70
0
10
20
30
40
50
60
70
% Filler by Wt. of 120 - 150 Pen. A.C.
% Filler by Wt. of 85 - 100 Pen. A.C.

The loess fillers again proved to be more effective in raising softening point than the limestone fillers. The difference is more apparent when incorporated with softer asphalt. (i.e. 120-150 pen. A.C.) In either case, small amounts of fillers have shown little effect on the softening point of the mastic. Up to about 25% filler content, the increase in softening point is approximately proportional to the amount of added filler; but for higher filler contents, the increase in softening point varies considerably.

In the case of mastic containing 85-100 pen. A.C., as the filler content increased, the softening point increased, very slowly up to 25%, and then, after 40%, it increased very rapidly and continuously to the maximum filler content studied.

As to the mastic containing 120-150 pen. A.C., the wide divergence begins at a filler content of 25%, while the loess fillers cause the mastic to have increasingly higher softening point at about the same rate as in 85-100 pen. A.C., the softening point of the mixtures containing limestone fillers increase at a very uniformly low rate up to about 70% filler content.

Though the fine-grained loess fillers are more effective in raising the softening point of the asphalt cement, the finer grade of the fillers (-# 325) are found less effective with respect to softening point increase to than that of coarser grade ones (-# 200) in all cases.

This indicates that the particle size or specific area of a filler is not the sole factor that determines the effectiveness in softening point increase when added to an asphalt cement, nor is the bulk density.

From the pratical point of view, the loess fillers would be regarded as a less desirable material when used as filler in asphaltic mixtures because of the possible high viscosity of the mastic at mixing temperatures due to its high softening point sensitivity.

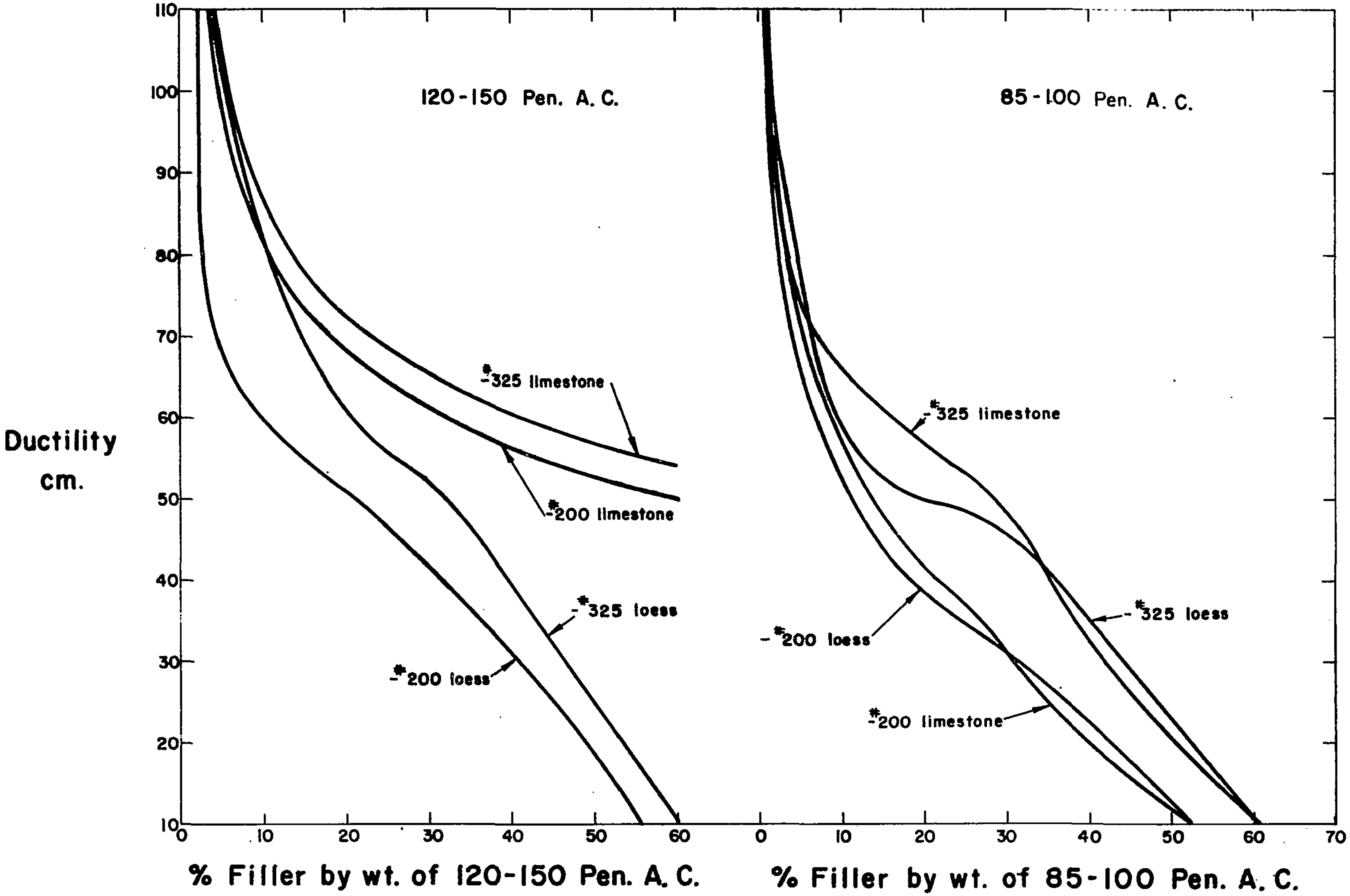
Ductility vs. Filler Content

Fig. 4 shows ductility in centimeters at 77 °F, 5 cm/min. versus filler content by weight of 120-150 pen. A.C. and 85-100 pen. A.C.

In all cases, as the filler content increases, the ductility decreases, very rapidly at first and then more slowly.

In the case of the mastics containing 85-100 pen. A.C., with all of the fillers tested the ductility rapidly decreases from 150+ to the 55-65 cm. range at 10% filler content. As for mastics containing 120-150 pen. A.C., ductilities are reduced to 80-90 cm. range at 10% filler content, with the exception of -# 200 loess which drops very rapidly to 60 cm. The loess mastics continued to lose ductility rapidly as the filler content was increased, a portion of the curve, over the

Fig. 4. Relationship between ductility of the mastics and percent filler by weight


120-150 Pen. A. C.
85-100 Pen. A. C.
Ductility cm.
110
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
-#325 limestone
-#200 limestone
-#325 loess
-#200 loess
-#325 limestone
-#325 loess
-#200 loess
-#200 limestone
% Filler by wt. of 120-150 Pen. A. C.
% Filler by wt. of 85-100 Pen. A. C.

range of 20-30% filler content, showed a tendency to flatten out. The limestone gives a similar result in 85-100 pen. A.C. as does the loess, but behaves very differently in 120-150 pen. A.C., leveling out quite appreciably and being much less critical in excess of 60% filler content by weight of asphalt cement.

This entirely different behavior of limestone dust with 120-150 pen. A.C. is also found in the cases of penetration as well as in viscosity studies.

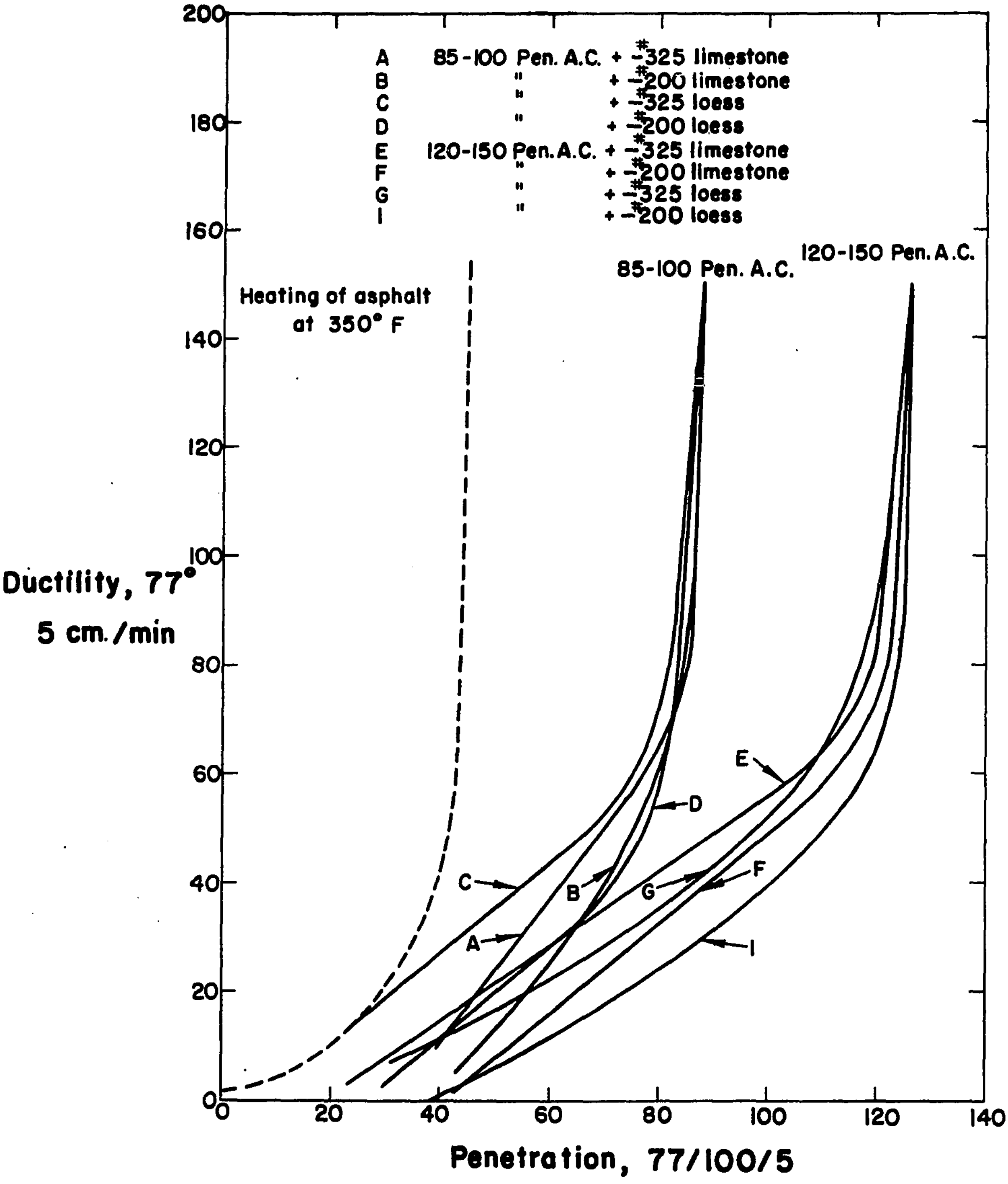
It is seen that the loess fillers are more effective in reducing ductility of the mastic than limestone fillers and that, for both loess and limestone dust, the finer grade fillers (-# 325) always result in higher ductility values for any filler content over entire range studied. It is also noted that the softer grade asphalt (120-150 pen. A.C.) tends to have less effect on ductility for a given filler and at a given filler content, even though both 120-150 pen. A.C. and 85-100 pen. A.C. have shown a ductility of 150+ at zero dust content.

Penetration vs. Ductility

Fig. 5 shows the relationship between penetration and ductility of the mastics, both at 77 °F.

Generally speaking, the lower the penetration of an

Fig. 5. Relationship between penetration and ductility of the mastics


A 85-100 Pen. A.C. + -#325 limestone
B " + -#200 limestone
C " + -#325 loess
D " + -#200 loess
E 120-150 Pen. A.C. + -#325 limestone
F " + -#200 limestone
G " + -#325 loess
I " + -#200 loess
Heating of asphalt at 350° F
85-100 Pen. A.C.
120-150 Pen. A.C.
A
B
C
D
E
F
G
I
Ductility, 77° 5 cm./min
Penetration, 77/100/5
0
20
40
60
80
100
120
140
160
180
200

asphalt cement at a given temperature, the lower will be its ductility at that temperature. There is, however, no direct relationship between these two properties. This is especially true when the asphalt has been subjected to successive heating to temperatures at which incipient polymerization may take place, as the ductility will decrease much more rapidly than the penetration. For comparative purposes, the penetration--ductility relationship of an asphalt under the effect of heat is also shown. (72, p. 118)

It is interesting to note that, as far as penetration and ductility are concerned, the effect of adding filler to an asphalt cement appears similar to that of heating, especially in small filler content range.

Since same scale was used in plotting penetration and ductility, it is readily seen that in all cases, the changes in ductility due to addition of filler is much more rapid than that in penetration, especially when the filler content is below 20%.

Conclusion can be drawn that, when carefully controlled, the mastic is more sensitive to ductility test than to penetration test. It is therefore suggested that the ductility test should be used to evaluate different mineral powders as fillers in asphaltic mixtures, especially at low filler contents.

It is also seen that, for a given penetration, the finer grade fillers (-# 325) yield higher ductility values than that of the coarser grade fillers (-# 200). This appears very significant from a practical point of view.

Penetration vs. Softening Point

Penetration-softening point relationships have been used by many asphalt technologists in evaluating susceptibility of asphalts.

The penetration-softening point relationships of asphalts were extensively studied by, among many other, Kinnaird (42, 43), Saal (66, 67) and Pfeiffer and Van Doormaal (58).

Kinnaird has found that:

(a) When the lines connecting the log softening point and log penetration of all asphalts from the same orgin are drawn it was found they all pass through the same common point.

(b) Similar data on bitumens of other origin yield lines that fall at other points; the various points for various "same stock" bitumens "are all on a fixed curve."

From this he was able to deduce the interesting relationship between penetration and softening point:

$$P_{77} = F \left[\frac{S.P.-77}{100} \right]^{-4.25/\sqrt[3]{F}}$$

where P_{77} = penetration in 10^{-2}mm at 77 ^{o}F/100 gr./5 sec.

F = characterizing factor

S.P.= R. and B. softening point in ^{o}F

The characterizing factor has been found to be closely related to the percentage of paraffienic constituents in the bitumen, and thus can be used to classify asphalts into groups that are similar in composition.

Saal, in a study of various methods of absolute viscosity determinations, found the following relationships for asphalts of Newtonian type:

(a) Between penetration and absolute viscosity when determined at the same temperature:

$$\eta = \frac{5.9 \times 10^{9}}{(\text{penetration})^{1.93}} \text{ poises}$$

A revised equation (108) states that:

$$\eta = \frac{1.58 \times 10^{10}}{(\text{penetration})^{2.16}} \text{ poises}$$

This formula can also be partly derived theoretically. The following is then obtained:

$$al + 1/2\ bl^{2} = \frac{Pt}{\eta}$$

in which:

η = absolute viscosity in poise

P = load of the needle in dynes

t = duration of penetration in seconds

l = penetration in centimeters

a,b = constants for needle resistance.

(b) Viscosity at the R and B softening point:

$$\eta = 1.2 \times 10^4 \quad \text{poises}$$

In an attempt to characterize asphaltic bitumens by the values found for the penetration tests at different temperatures and for the R and B softening point, Pfeiffer and van Doormaal proposed an penetration index (P.I.), which can be calculated from one penetration value and the R. and B. softening point, by the following relationship, or from a penetration index nomograph:

$$\frac{20 - \text{P.I.}}{10 + \text{P.I.}} \; \frac{1}{50} = \frac{\log 800 - \log \text{Penetration at } T^{o}C}{\text{Softening point } ({}^{o}C) - T^{o}C}$$

This deduction was based on:

(a) The relation that

$$\log \text{penetration} = K + \alpha t$$

where α is a characteristic of the temperature susceptibility; k is the log of the penetration at $0^{o}C$ and t is the temperature in ^{o}C at which the penetration is determined.

(b) The penetration at the R and B. softening point amounts to about 800, a value which is derived from results found by Saal and is confirmed by experiment by Pfeiffer and van Doormaal for asphalts of both purely viscous type and elastic type.

According to P.I. values, asphalts are grouped as follows:

(a) For highly temperature-susceptible materials, the P.I. varies from about -1 to lower. Most sol asphalts, coal-tar pitches, and those from cracking processes are in this group.

(b) "Normal types" range from -1 to +1. This range includes most viscoelastic asphalts.

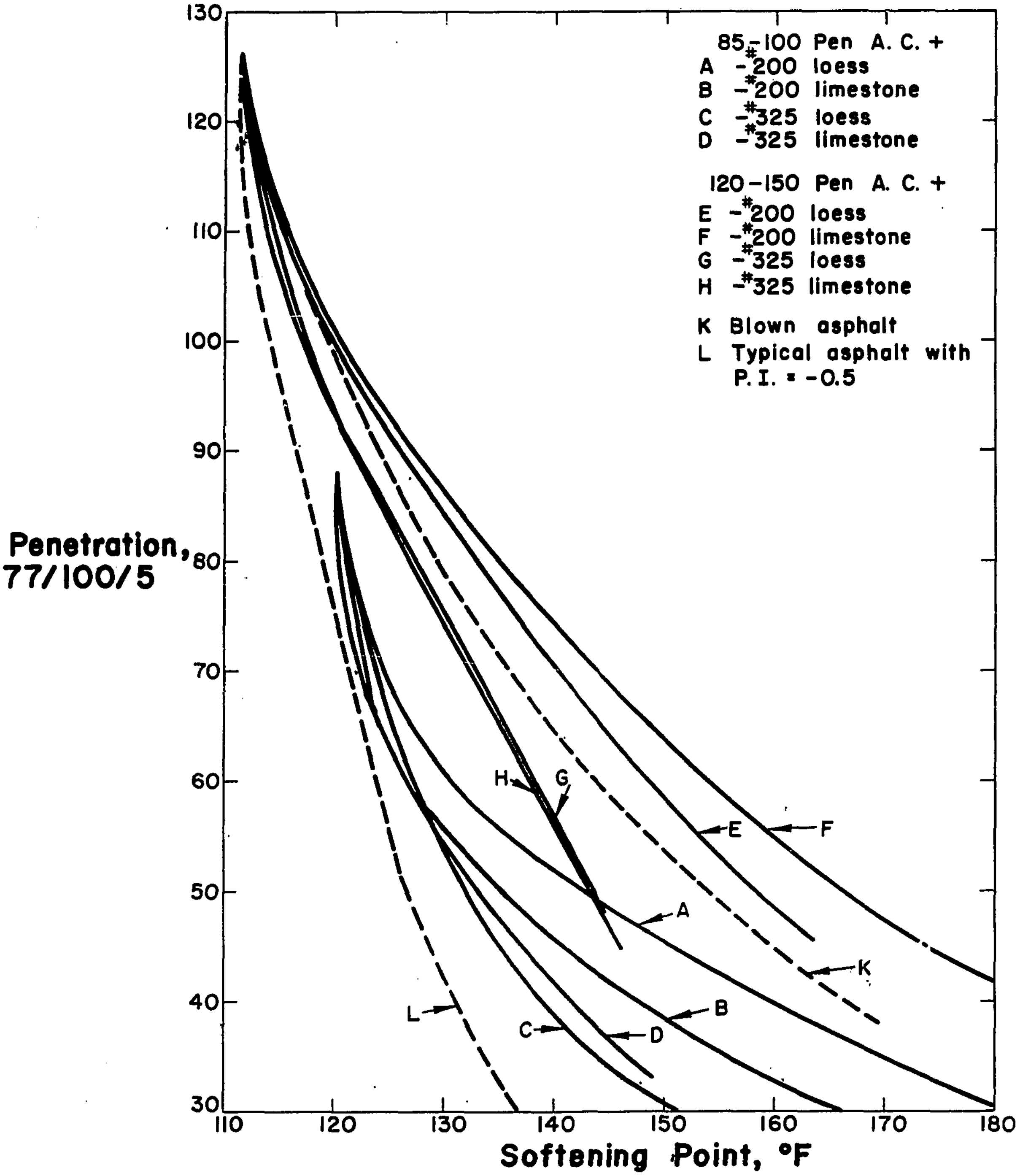
(c) Low temperature-susceptible asphalts of the gel type range in values from +1 to as high as 6 or 7. Most of blown asphalts belong to this type.

Fig. 6 shows the penetration-softening point relationships for the eight filler-asphalt systems studied. For comparative purposes, a typical penetration-softening point curve for asphalts with P.I. of -0.5 is plotted according to Pfeiffer and van Doormaal's nomograph. A blowing curve from a comparable straight-run asphalt to a higher softening point blown asphalt is also plotted. (10, p. 411)

It is noted that the addition of filler has about the same effect on penetration-softening point as does blowing the asphalt.

It is also seen that the rate of change of penetration

Fig. 6. Relationship between penetration and softening of the mastics


85-100 Pen A. C. +
A -#200 loess
B -#200 limestone
C -#325 loess
D -#325 limestone
120-150 Pen A. C. +
E -#200 loess
F -#200 limestone
G -#325 loess
H -#325 limestone
K Blown asphalt
L Typical asphalt with P. I. = -0.5
Penetration, 77/100/5
Softening Point, °F
130
120
110
100
90
80
70
60
50
40
30
110
120
130
140
150
160
170
180
A
B
C
D
E
F
G
H
K
L

is greater than that of softening point at low filler content and that the rate of the change of softening point becomes greater at higher filler content.

There is no indication as to which type of filler is more effective in changing the asphalt penetration-softening point relationships, but it appears that the coarser grade fillers cause more deviation from the pure asphalt than do the finer grade ones.

If using temperature susceptibility as a criteria in evaluating fillers in an asphaltic mix, the best filler may be considered to be the one which will yeild a mastic that has the highest softening point for a given penetration; and by this concept, the -# 200 sieve fillers would be considered superior to -# 325 sieve fillers and the mastics thus prepared would be superior to blown asphalts.

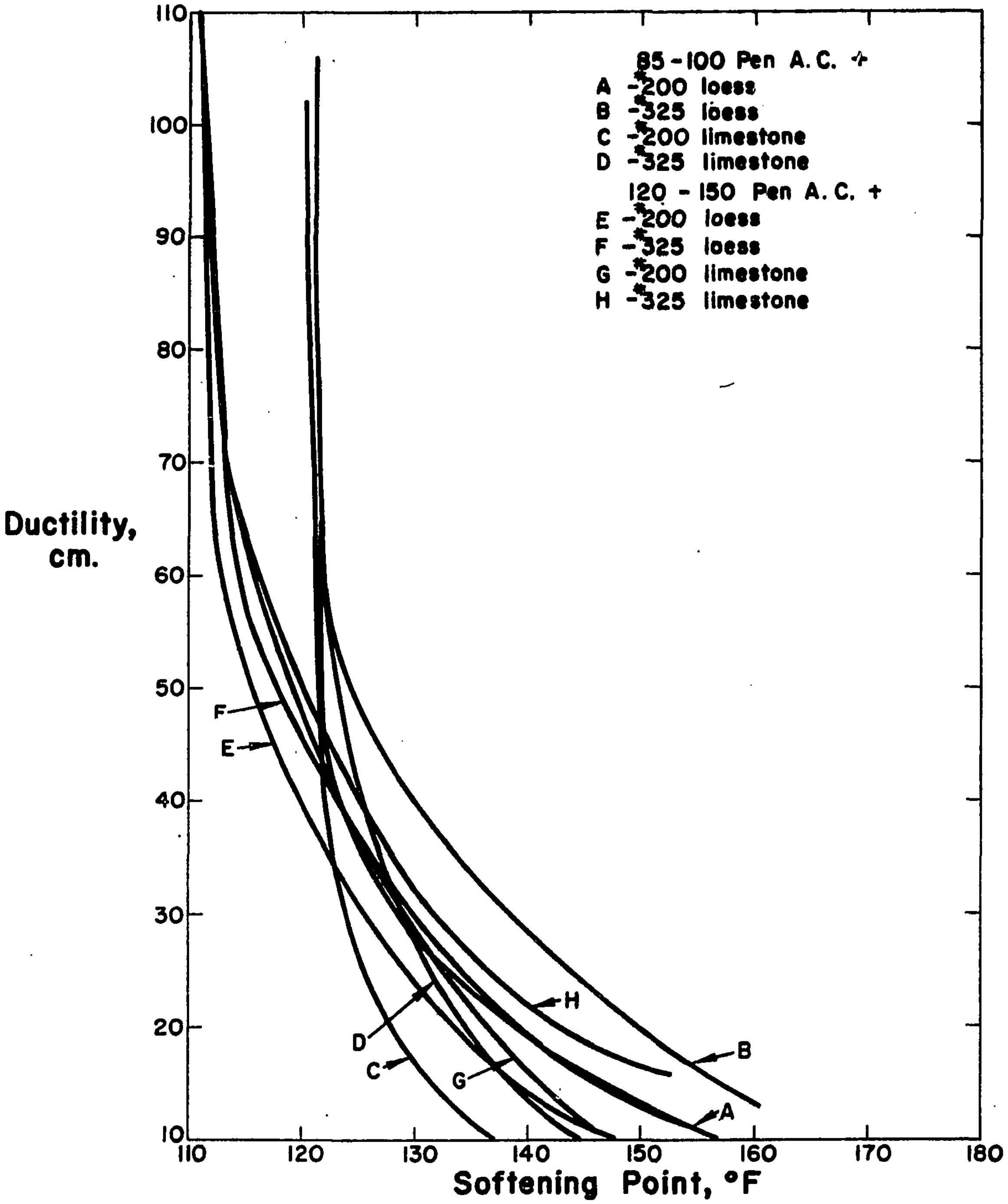
Ductility vs. Softening Point

Fig. 7 shows the relationship between ductility at 77 °F, 5 cm/min., and softening point, °F, of the mortar. It is seen from the curves that:

(a) The ductility drop is much more rapid than the softening point rise, especially at low filler content.

(b) At higher filler contents, when ductility drops to below 40 cm, the softening point of the original asphalts cannot be considered a guide to the softening point of the mastic.

Fig. 7. Relationship between ductility and softening of the mastics


85-100 Pen A.C. +
A -*200 loess
B -*325 loess
C -*200 limestone
D -*325 limestone
120 - 150 Pen A.C. +
E -*200 loess
F -*325 loess
G -*200 limestone
H -*325 limestone
Ductility, cm.
110
100
90
80
70
60
50
40
30
20
10
Softening Point, °F
110
120
130
140
150
160
170
180
A
B
C
D
E
F
G
H

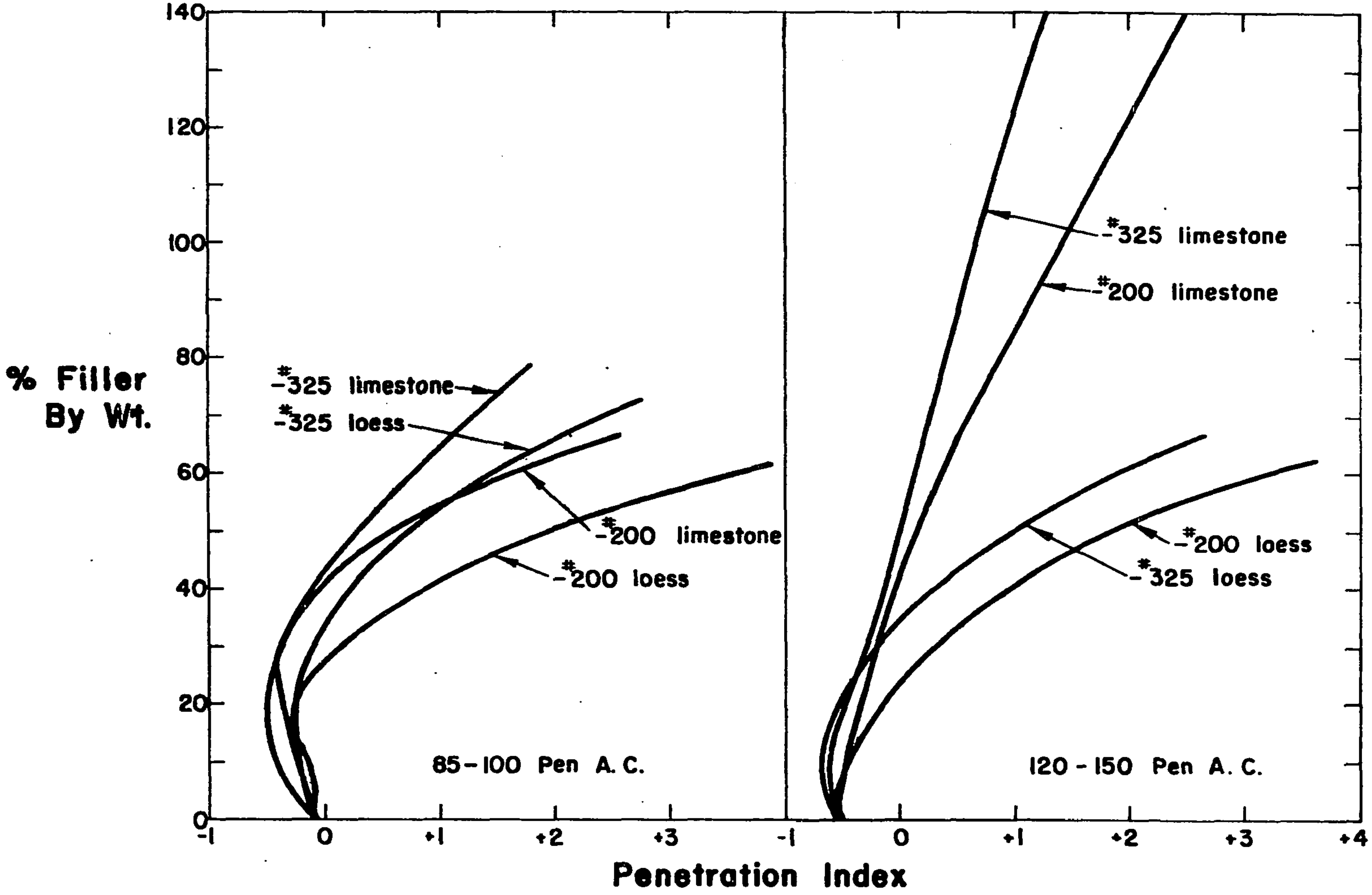
(c) For a given softening point, the mastic with finer grade fillers gives higher ductility values in all cases. This is another property of the mastic that depends more on filler size than on filler type or asphalt type.

Penetration Index vs. Filler Content

For a great many technical applications the changes in consistency between 0°C and the temperature of softening point form a highly important and much discussed quality consideration in judging the utility of an asphaltic bitumen. This property of temperature susceptibility as reflected by penetration index proposed by Pfeiffer and van Doormaal has been related to various mastic properties of different fillers and filler contents. Fig. 8 shows the relation between P.I. determined from the nomograph prepared by Pfeiffer and van Doormaal and filler content by weight.

In all cases the P.I. drops slightly at first and then, as the filler content in the mastic is increased, the P.I. increases and differences in the behavior of various fillers and asphalts become evident. The slight drop of P.I. at first is because the added filler at low filler content has more effect on penetration reduction than on softening point increasing. Referring to penetration-softening point curves (Fig. 6), it should be pointed out that this slight drop in P.I., seems to be the difference between the effect of adding

Fig. 8. Relationship between penetration index of the mastics and percent filler by weight



% Filler By Wt.
140
120
100
80
60
40
20
0
-1
0
+1
+2
+3
-1
0
+1
+2
+3
+4
Penetration Index
-#325 limestone
-#325 loess
-#200 limestone
-#200 loess
85-100 Pen A. C.
-#325 limestone
-#200 limestone
-#200 loess
-#325 loess
120 - 150 Pen A. C.

fillers to the asphalt and blowing asphalt.

It is noted that, as far as the increasing of P.I. is concerned, loess fillers are more effective than limestone fillers and coarser grade fillers are more effective than finer grade ones. As shown in Fig. 8, the percentages of various fillers required to increase the P.I. of mortar to +1 are:

	85-100 pen. A.C.	120-150 pen. A.C.
-# 200 loess	42%	42%
-# 325 loess	52%	50%
-# 200 limestone	54%	84%
-# 325 limestone	64%	122%

Since (a) P.I. can practically indicate the temperature susceptibility and the degree to which the rheological behavior of the mastic deviates from that of the purely viscous asphalt (or the degree of elasticity), (b) P.I. can be readily determined using the nomograph and well standardized and universally used tests, (c) it can indicate large differences among fillers, being independent of the asphalts used; it is suggested that it be used as a criterion in evaluating materials used as fillers in asphalt mixes.

Viscosity vs. Filler Content

The viscosity of a bituminous binder material is a measure of its flow characteristics, and affects to a considerable degree the performance of a bituminous paving mixture in which the binder is used. Viscosity may therefore be considered the most important single physical property of a binder.

Asphalt cements are supplied in various grades having, at normal temperatures, a wide range of consistency from fluid to hard and brittle. For different types of road construction, traffic and weather, a binder having suitable flow properties must be selected and controlled. In order that a bituminous material can be used successfully in paving mixture it must have the following rheological characteristics:

(a) It is capable of being sufficiently fluid to be pumped or sprayed, and to coat and wet a mineral aggregate.

(b) It is capable of becoming so viscous at high road temperatures (up to 140 °F) that the finished paving will resist deformation.

(c) It is capable of being so flexible at low road temperatures that the finished paving will resist fracture and disintegration.

The study of viscosity of asphalt has thus become one of the key tools in the recent efforts and advances in research or paving asphalts toward the "idealized asphalt" (47).

Though the absolute viscosity in poise has not been used in specifications for asphalt cements, the importance of using this unit in research has very well been recognized. In order to study the effect of various fillers to the viscosity of various asphalts at applying temperatures (225 ^{o}F to 325 ^{o}F), the absolute viscosity of mastics were determined by Brookfield viscometer. The viscosity-filler content relationships are shown in Fig. 9 and Fig. 10.

By comparing the curves, the followings are noted:

(a) In all cases, the viscosity of the mastic is increased with increasing filler content. The viscosity of the mastic is proportional to amount of added filler up to about 10%. The points of inflection of all curves occur at about 40-50% filler content, from where the rate of change of the viscosity due the added filler increases rapidly throughout the range studied.

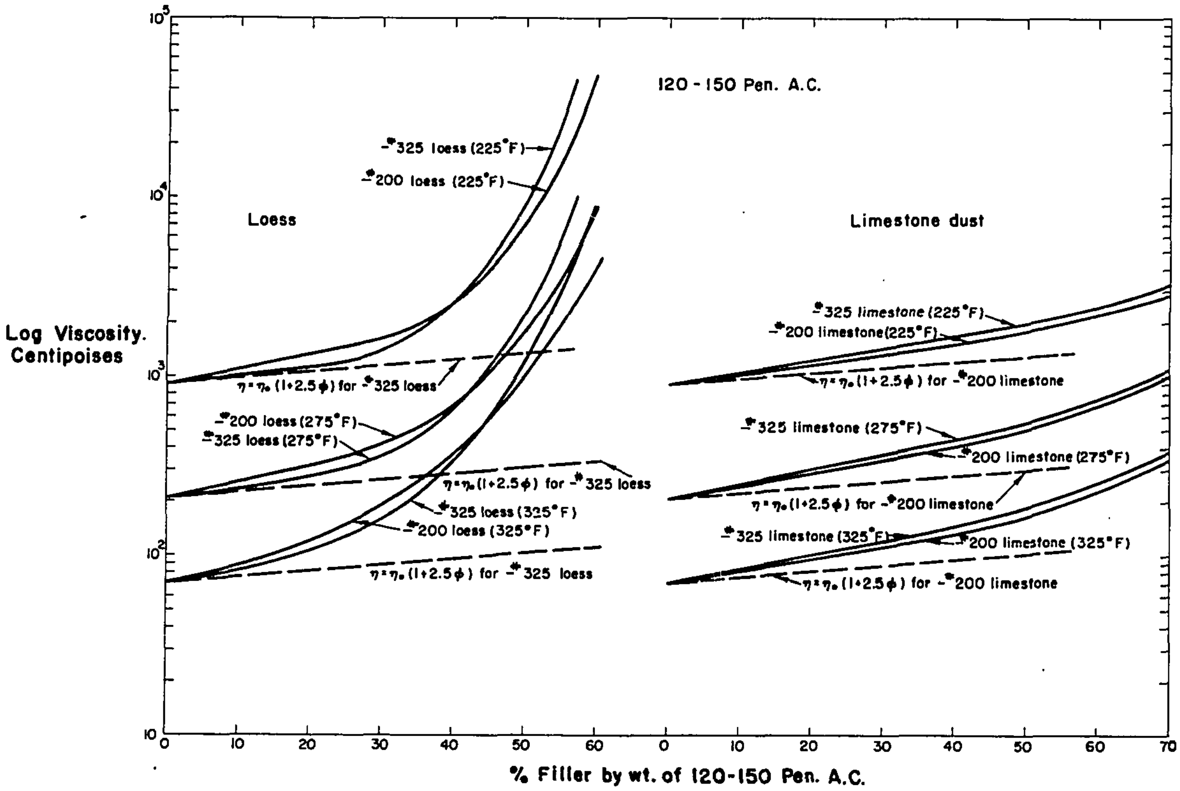
(b) Einstein's equation (25):

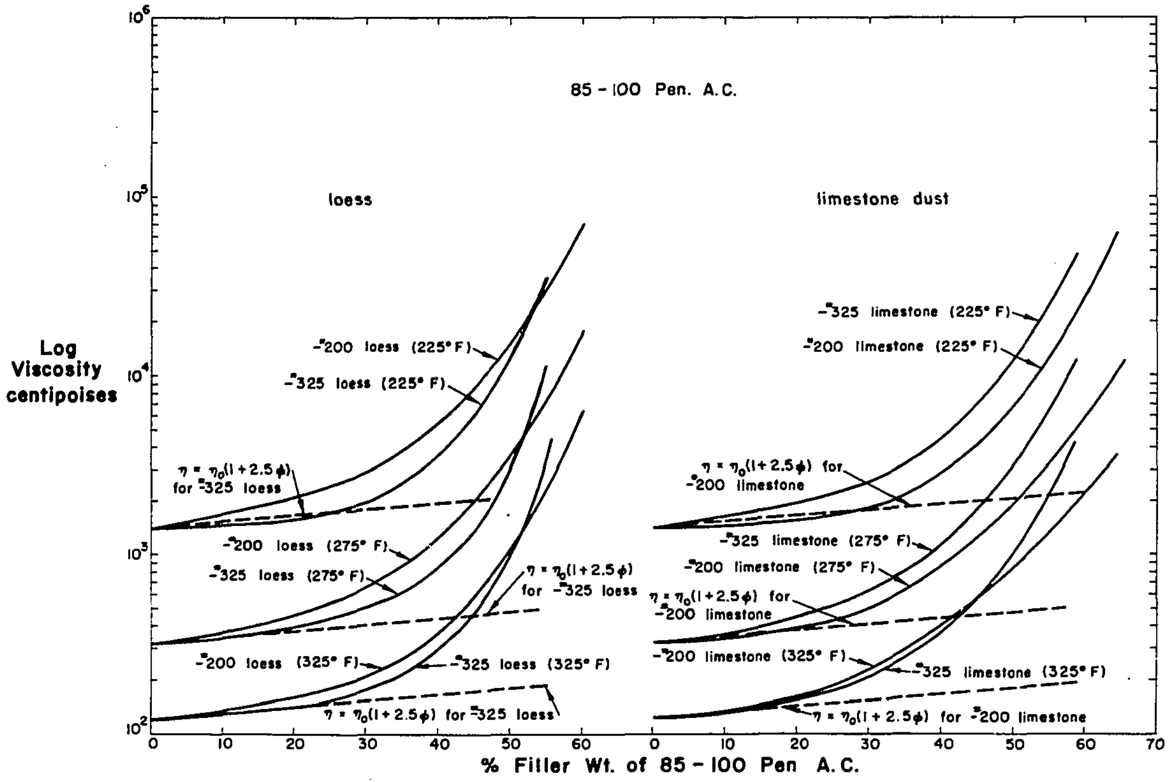
$$\eta = \eta_o(1 + 2.5\phi)$$

holds true only at very low filler contents (0 - 2%). This is probably due to the fact that the mastic studied is not a true colloidal suspension system as the particles are fairly large and due to the more sluggish movement of the particles and the frictional forces between particles at more compacted high filler contents. Also, Einstein's equation assumes the solid particles are of "small rigid spheres" which is not likely the case in filler-bitumen system. In this equation, η_o is the viscosity of the dispersion medium; η, the viscosity of the suspension, and the ϕ volume fraction occupied by solid

Fig. 9. Plot of log viscosity vs. percent filler by weight for 120-150 pen. A.C.

Fig. 10. Plot of log viscosity vs. percent filler by weight for 85-100 pen. A.C.


120 - 150 Pen. A.C.
Loess
Limestone dust
Log Viscosity. Centipoises
10
10^2
10^3
10^4
10^5
-#325 loess (225°F)
-#200 loess (225°F)
η = η₀ (1+2.5φ) for -#325 loess
-#200 loess (275°F)
-#325 loess (275°F)
η = η₀ (1+2.5φ) for -#325 loess
-#325 loess (325°F)
-#200 loess (325°F)
η = η₀ (1+2.5φ) for -#325 loess
-#325 limestone (225°F)
-#200 limestone (225°F)
η = η₀ (1+2.5φ) for -#200 limestone
-#325 limestone (275°F)
-#200 limestone (275°F)
η = η₀ (1+2.5φ) for -#200 limestone
-#325 limestone (325°F)
-#200 limestone (325°F)
η = η₀ (1+2.5φ) for -#200 limestone
0
10
20
30
40
50
60
0
10
20
30
40
50
60
70
% Filler by wt. of 120-150 Pen. A.C.


85 - 100 Pen. A.C.
loess
limestone dust
Log Viscosity centipoises
10^2
10^3
10^4
10^5
10^6
-#200 loess (225°F)
-#325 loess (225°F)
η = η₀(1+2.5φ) for -#325 loess
-#200 loess (275°F)
-#325 loess (275°F)
η = η₀(1+2.5φ) for -#325 loess
-#200 loess (325°F)
-#325 loess (325°F)
η = η₀(1+2.5φ) for -#325 loess
-#325 limestone (225°F)
-#200 limestone (225°F)
η = η₀(1+2.5φ) for -#200 limestone
-#325 limestone (275°F)
-#200 limestone (275°F)
η = η₀(1+2.5φ) for -#200 limestone
-#200 limestone (325°F)
-#325 limestone (325°F)
η = η₀(1+2.5φ) for -#200 limestone
0
10
20
30
40
50
60
0
10
20
30
40
50
60
70
% Filler Wt. of 85 - 100 Pen A.C.

in the suspension.

(c) The degree of relative effectiveness of different types of fillers on the viscosity of the mortar is found less evident in more viscous medium (85-100 pen. A.C.) than in less viscous medium (120-150 pen. A.C.).

(d) The ratio η/η_0 is found to vary with both the viscosity of the medium (η_0) and the fineness of the particle size. For limestone fillers, the finer the particle size of the filler, the greater is the ratio; but this holds true only at higher filler contents (about 40-50%) for loess fillers.

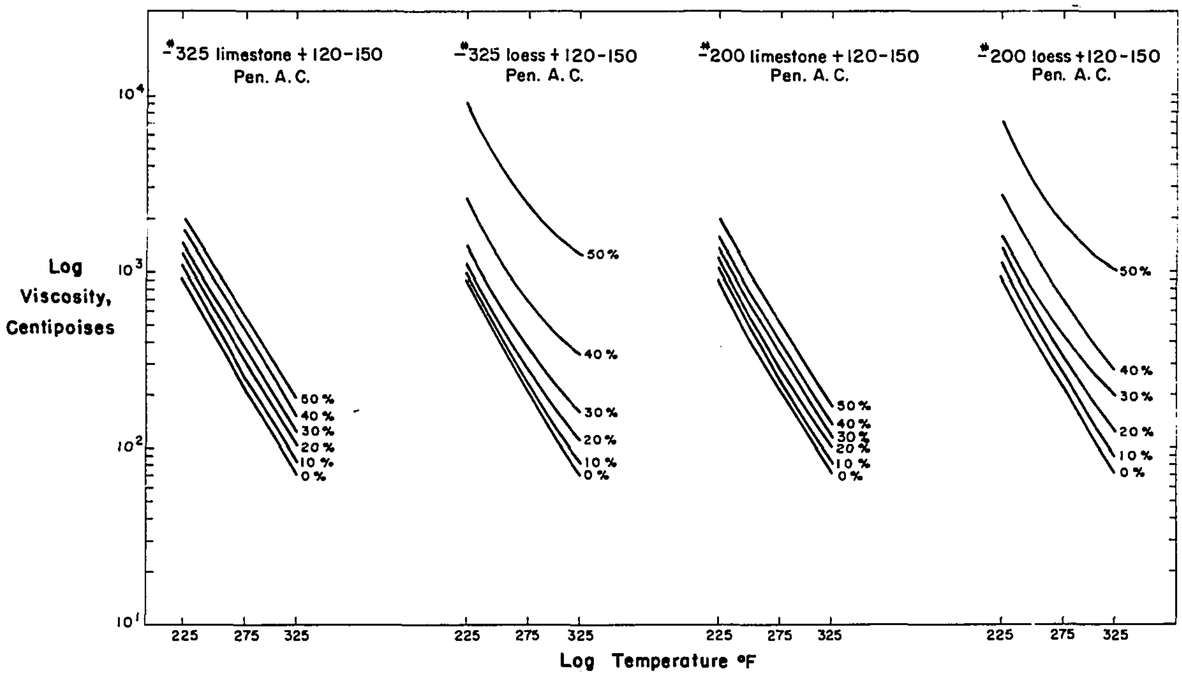
Log Viscosity vs. Temperature

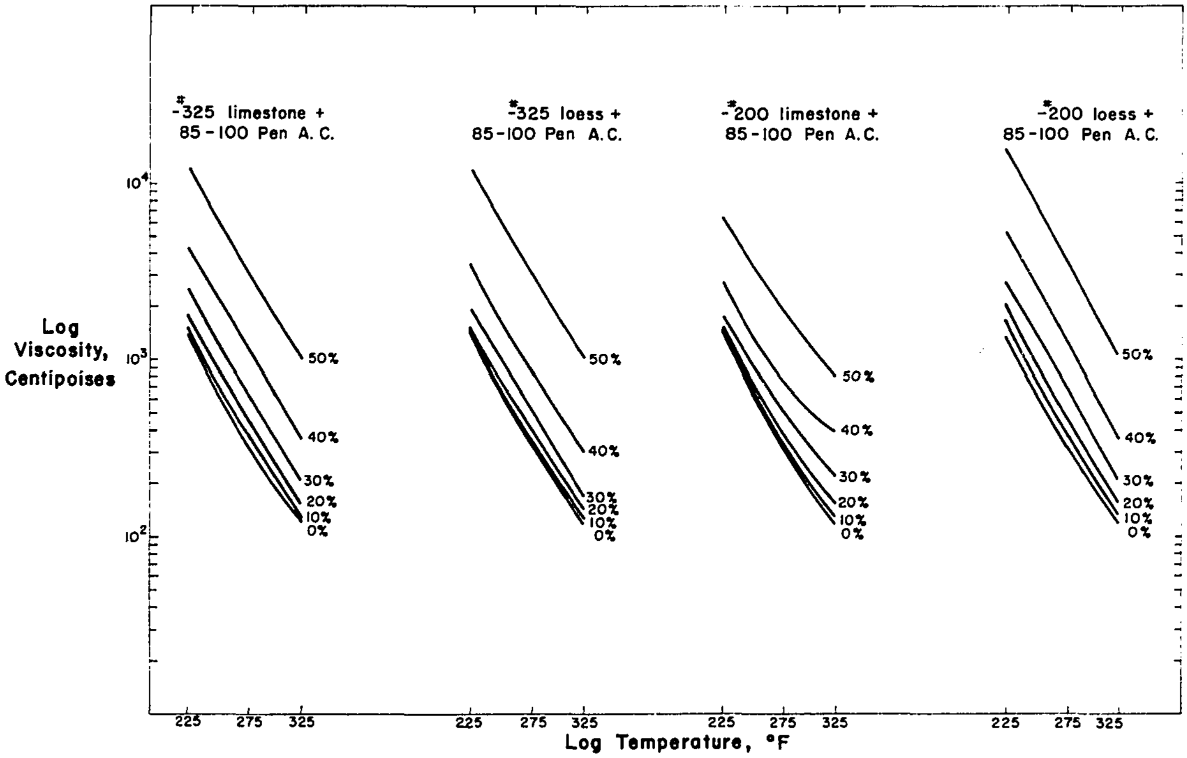
Fig. 11 and 12 show the relationship of viscosity and temperature of mastic for various filler contents. It is the characteristics of bituminous materials that the viscosity falls very rapidly as the temperature rises and that the change in viscosity with temperature is greater at low temperatures than at high temperatures.

The relationship of the change of viscosity of bitumen with temperature has been studied and suggested by various investigators (53). However, it has been found that the viscosity vs. temperature relationships of bitumens are complex. At a fixed temperature it varies with the type, quality and composition, and no one single equation has been found that is of pratical value over a wide temperature range.

Fig. 11. Plot of log viscosity vs. temperature for mastics containing 120-150 pen. A.C.

Fig. 12. Plot of log viscosity vs. temperature for mastics containing 85-100 pen. A.C.


-#325 limestone + 120-150 Pen. A.C.
-#325 loess + 120-150 Pen. A.C.
-#200 limestone + 120-150 Pen. A.C.
-#200 loess + 120-150 Pen. A.C.
Log Viscosity, Centipoises
10^4
10^3
10^2
10^1
50%
40%
30%
20%
10%
0%
225
275
325
Log Temperature °F


-#325 limestone + 85-100 Pen A.C.
-#325 loess + 85-100 Pen A.C.
-#200 limestone + 85-100 Pen A.C.
-#200 loess + 85-100 Pen A.C.
Log Viscosity, Centipoises
10^4
10^3
10^2
50%
40%
30%
20%
10%
0%
225
275
325
Log Temperature, °F

Among those findings two are commonly recognized:

(a) When the logarithm of the viscosity in poises is plotted against the logarithm of the temperature in degrees $^{\circ}F$ a substantially linear relationship is obtained over a wide range of temperature. (32, 52)

(b) When log-viscosity is plotted against ordinary temperature (in either $^{\circ}C$ or $^{\circ}F$), straight-line relationship has been found between processing temperatures where the asphalts have viscosities ranging from 1 to 5 poises and at service temperatures (59 $^{\circ}F$ to 95 $^{\circ}F$) where the asphalts have viscosities ranging from 10^6 to 10^{10} poises. (69)

The asphalt cements and mastics studied show slight deviations from both of these two relationships. However, the effects of adding mineral fillers are apparent:

(a) The viscosity increase due to the addition of 1% filler is greater at lower temperatures than at higher temperatures and is greater at higher filler contents than at lower contents.

(b) Generally speaking, the finer grade fillers are more effective in increasing viscosity than the coarser grade fillers, and the more viscous binder (85-100 pen. A.C.) increases its viscosity more rapidly for a given amount of filler added than does the less viscous binder. (120-150 pen. A.C.)

(c) Loess fillers show greater relative effectiveness in increasing the viscosity of the mastic than do the limestone fillers, except for -# 325 limestone filler, which yields mastics of similar viscosity to -# 325 loess in 85-100 pen. A.C. at equal filler contents.

(d) With the exception of loess in 120-150 pen. A.C., temperature susceptibility of viscosity of the asphalt shows little, if any, change due to the addition of filler. This temperature susceptibility can be determined either in terms of asphalt viscosity index (69) or by logarithmic temperature coefficient of viscosity n (94, 115).

$$\text{Asphalt viscosity index} = 100 \left[(\eta_a/\eta_b)^{1/ta-tb} - 1 \right]$$

where η_a and η_b are viscosities at two temperatures ta and tb (deg. Cent.)

$$n = \frac{\log \eta_1 - \log \eta_2}{\log T_2 - \log T_1}$$

where η_1 and η_2 are the viscosities of the mortar at T_1 and T_2 (^{o}F).

(e) In a general trend, the relative effectiveness of the filler in increasing the viscosity of the filler-asphalt mastic increases with the decreasing bulk density and increasing kerosene voids of the filler.

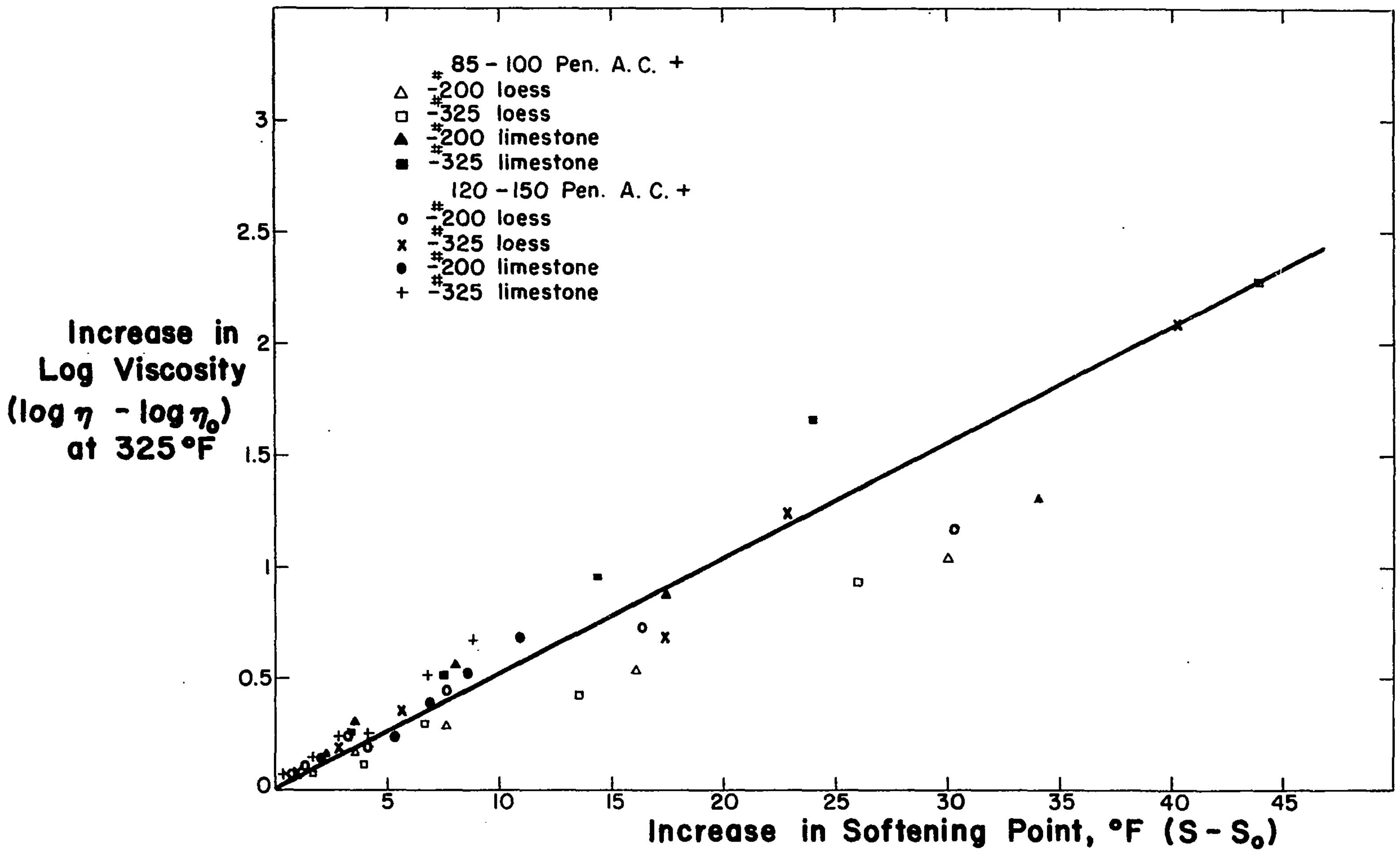
Increase in Viscosity vs. Increase in Softening Point

Fig. 13 shows the relationship between increase in viscosity at 325 °F and increase in softening point. A straight line is obtained between the increase in log viscosity and the increase in softening point in degree. The straight line passes through the origin and is independent of the asphalt type, of filler and filler concentration. For the asphalts and fillers studies, an increase in softening point of 10 °F corresponds to an increase in log of 0.5. Similar results can be seen by comparing softening point vs. filler content curve and viscosity versus filler content curves. (Fig. 3, Fig. 9 and Fig. 10) Thus it can be stated that the changes in softening point definitely reflect changes in viscosity and this relationship makes the softening point test a more valuable method in evaluating mineral filler used in bituminous paving mixtures.

Percent Filler in Suspension

In order for the filler to be effective in modifying the rheological properties of asphalt both during processing temperatures and at service temperatures, the filler must be capable of being suspended in the asphalt not only at high mixing temperatures while the asphalt is fluid, but also remaining in suspension at road temperatures while the asphalt is very viscous.

Fig. 13. Relationship between increase in viscosity and increase in softening point of the mastics


85 - 100 Pen. A. C. +
△ -#200 loess
□ -#325 loess
▲ -#200 limestone
■ -#325 limestone
120 - 150 Pen. A. C. +
o -#200 loess
x -#325 loess
● -#200 limestone
+ -#325 limestone
Increase in Log Viscosity (log η - log η₀) at 325 °F
0
0.5
1
1.5
2
2.5
3
5
10
15
20
25
30
35
40
45
Increase in Softening Point, °F (S - S₀)

For proper design of an asphalt paving mixture it is necessary to know quantitatively both what amount of filler and which portion of filler will remain in suspension in a bituminous mastic over this wide temperature range and the long pavement life period.

Theoretically the amount of filler in suspension in the continuous phase of bitumen at any temperature is related to: (a) the viscosity of the bitumen (b) the specific gravity of the bitumen (c) the specific gravity of the filler and (d) the particle size and particle shape of the filler.

Since the dominant factor is the viscosity of the asphalt cement which decreases greatly with the increase of temperature, and since the amount of filler in suspension is inversely proportional to the viscosity of the asphalt cement, it is the amount of filler in suspension at the higher temperatures in process of preparing the mixture that is primarily concerned.

The conventional asphaltic mixtures are prepared between 275 °F and 325 °F, thus it is the amount of filler in suspension at this temperature range that was of interest in this investigation.

Colloidal suspension of filler in asphalt has been suggested by many investigators. But so far the term has been used very loosely and no one seems to know which particles are colloidal in bitumen. For the purpose of this investigation, as well as practical considerations, colloidal mineral particles are defined as those particles that remain in suspension in asphalt for 24 hours at a temperature of 325 °F which corresponds to a viscosity of about 1-2 poises for ordinary penetration grade asphalt.

In Tables 3 - 10, the percent filler in suspension was calculated from the sum of weight of fillers in top and middle layers divided by the total weight of fillers found in all three layers expressed in percentage.

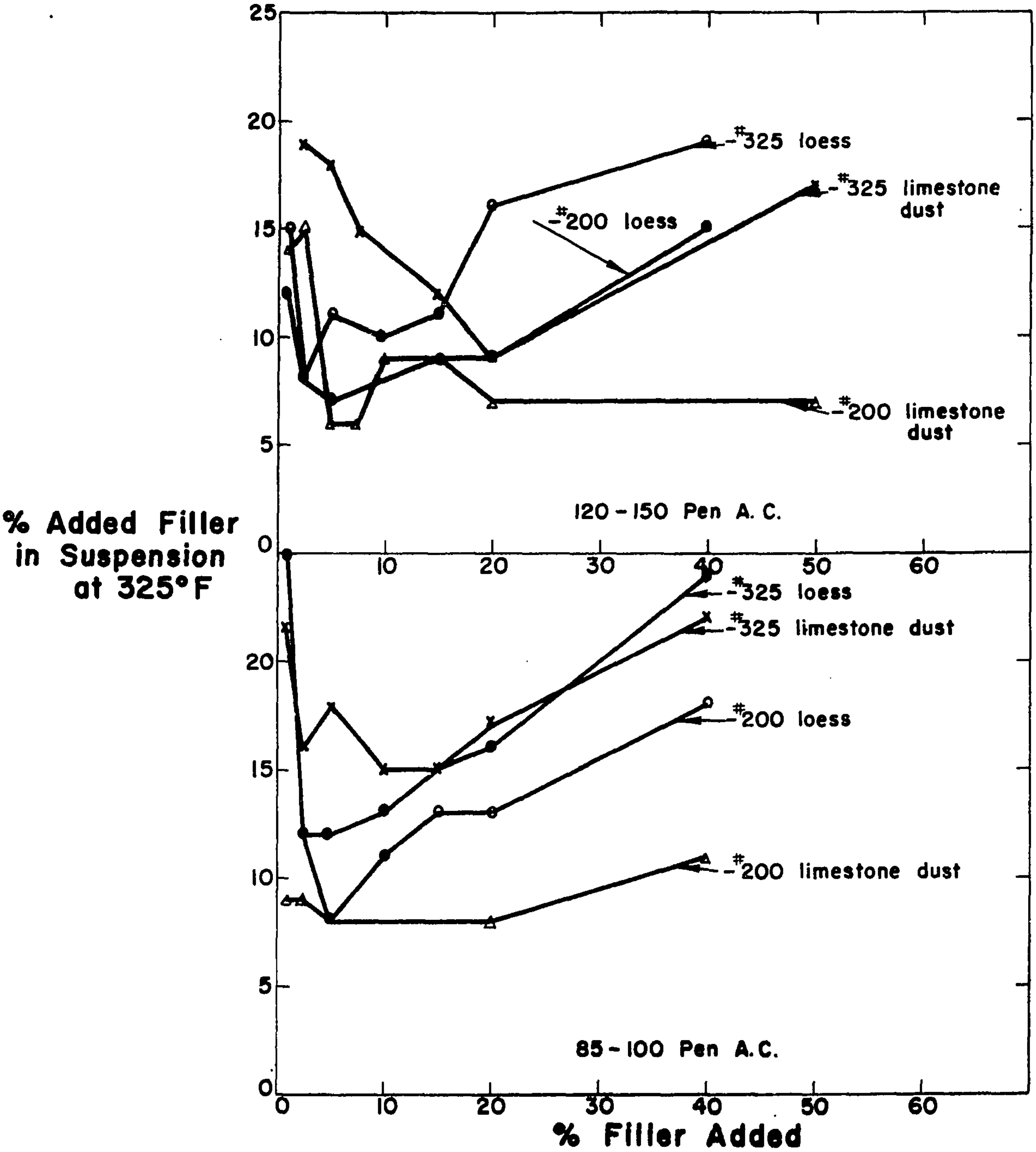
Fig. 14 shows the relationship between percent filler in suspension and the percent filler added to the mixture.

Various efforts had been made to determine the filler content in each layer after 24 hours settlement, among those included ignition method and the use of C-H analyzer.

The inconsistent results and large errors at low added filler contents have always been the problems. The reason of this was believed to be due to the small sample size, inhomogeneuity of the sample in comparison to the sample size and the inconsistent mineral composition in comparison with the sample size. Even for the finally adopted method, Soxhlet extraction, the errors were still found at low filler contents. Therefore, in drawing conclusions here, the results from mixtures with below 5% added filler contents were disregarded.

(a) Between 5% to 20% added filler, average percent filler in suspension or percent colloidal suspension in 120-

Fig. 14. Relationship between percent added filler and percent filler in suspension


% Added Filler in Suspension at 325°F
% Filler Added
120 - 150 Pen A. C.
-#325 loess
-#325 limestone dust
-#200 loess
-#200 limestone dust
85 - 100 Pen A.C.
-#325 loess
-#325 limestone dust
-#200 loess
-#200 limestone dust
0
5
10
15
20
25
0
10
20
30
40
50
60

150 pen. A.C. are:

-# 200 limestone	7%
-# 200 loess	8%
-# 325 loess	12%
-# 325 limestone	14%

Percents filler in colloidal suspension in 85-100 pen. A.C. are:

-# 200 limestone	8%
-# 200 loess	11%
-# 325 loess	14%
-# 325 limestone	16%

(b) The average particle sizes corresponding to the amount of suspension by comparing with Fig. 1 are:

In 120-150 pen. A.C. (viscosity at 325 °F = 70 c.p.)

loess	0.3μ
limestone	5-8μ

In 85-100 pen. A.C. (viscosity at 325 °F = 120 c.p.)

loess	0.7μ
limestone	6-9μ

Assuming the filler particles are all spherical, theoretically particle sizes in colloidal suspension in 120-150 pen. A.C., as defined by this paper, are, calculated according to Stokes' law to be

For loess $r = 2.62\mu$ $d = 5.24\mu$

For limestone $r = 2.55\mu$ $d = 5.10\mu$

Theoretical particle sizes that will be in colloidal suspension in 85-100 pen. A.C. are

For loess $r = 3.44$ $d = 6.88$

For limestone $r = 3.34$ $d = 6.68$

r and d being the radius and diameter of the filler particle.

(c) Above 20% added filler, the percent filler in colloidal suspension increases continuously with the increasing filler content because of rapidly increased viscosity of the suspension system due to added filler. At 40% added filler, the particle sizes that are capable of being maintain in suspension are: 1.5 to 3.2μ for loess and 6-12μ for limestone.

(d) It is seen from above that the particle size is not the only factor in determining the amount of filler in suspension even the viscosity of bitumen is fixed. Other factors are the specific gravity of the filler, the particle shape of the filler and surface attraction between the filler and the bitumen which relates to the adsorption, surface tension, etc.

SUMMARY AND CONCLUSIONS

The relationships of asphaltic binder and filler, as expressed in terms of simple, universally adopted tests, were studied. Grades of asphalts and types of fillers were chosen with the consideration of both their popularities and their potentialities used in conventional high type asphaltic paving mixtures. Emphasis was placed on the filler particle size which has been included in current specifications and which could be used conveniently in defining the filler. Furthermore, most of the other important properties of filler such as specific surface area, voids, void diameter, degree of compaction, and bulk density as found by many other investigators, are directly or indirectly related to particle size. However, this does not imply that other filler properties, such as chemical reactivity, surface adsorption, surface absorption, surface texture, and particle strength etc., of the filler are less important. Mineral composition and particle shape of the fillers used in this investigation were studied, but due to limited information, no efforts were made to relate these properties to the mastic behavior. Consequently, more generalized postulation beyond the limits of this study is not warranted.

The investigation discloses that:

1. For all filler-asphalt mastics studied, as the filler content increases:

(a) Penetration decreases uniformly through out the filler contents studied.

(b) Softening point increases, first slowly, then very rapidly and shows large diversion among fillers.

(c) Ductility drops, very rapidly up to 10% filler content, and then flattens out.

(d) Viscosity at all temperatures increases, proportional to filler content up to about 10%, and rapidly and continuously after 40-50% filler.

(e) Penetration index decreases slightly at first, then increases at different rates continuously throughout the filler content range studied. This means that the temperature susceptibility decreases and consistency of the mastic increases.

(f) The softening point changes reflect the viscosity changes in a definite manner.

(g) For mastics of equal consistency or penetration, those containing softer asphalt and higher proportion of filler have the higher softening point and lower ductility.

2. For all mastics studied, the penetration and ductility are decreased, and the softening point and viscosity are increased, in addition to the increase of filler content, by the following factors, two or more of them may operate simultaneously: (a) increase in the fineness of the filler

or decrease in the particle size of the filler; (b) increase in the consistency of the asphalt; and (c) decrease in the specific gravity of the filler.

3. In more than one aspect, namely the penetration vs. ductility and the penetration vs. softening point relationships, the addition of filler simulates that of blowing or heating of asphalt.

4. As far as the relative effectiveness of the fillers in modifying the penetration, softening point, ductility, viscosity and temperature susceptibility properties of an asphalt is concerned, loess filler taken from mid-western Iowa is superior to limestone dust.

5. It appears that the relationships between the penetration index, ductility, softening point and viscosity of the mastic and the nature and proportion of the filler are most consistent and informative, thus, when very well controlled, they can be best adopted as evaluation tests for filler used in asphaltic paving mixtures.

6. Owing to the absence of any simple relationship between penetration, softening point and ductility of the filler-asphalt mastic, each of these properties must be determined if proper conclusions are to be deduced in evaluating mineral fillers used in asphaltic mixtures.

7. From 1, it is suggested that the function of filler in an asphaltic mix is to increase the viscosity of the

binder, reduce the temperature susceptibility and increase the plasticity of the mastic. The end effects on the paving mix are: (a) increases the stability or rigidity of the mix while still allowing it to retain the necessary flexibility; (b) reduces the amount of binder required as a filler of the voids in the aggregate; (c) enables a softer binder to be used without bleeding in summer and brittle in winter; and (d) gives toughness to the paving mix.

8. From 2 and 7, the essential properties of filler used in asphaltic paving mixture can be summarized as:

(a) To be able to become suspended in the binder, when the latter is in the fluid as well as the semi-solid or solid state.

(b) To be chemically inert.

9. With regard to the suspendibility of filler in asphalt, two important properties of the filler should be emphasized: (a) the fineness of the particle, and (b) the specific gravity of the filler. The joint result is: the smaller the particle size, the lower the specific gravity the longer or the more will the particles remain in the suspension.

It has been shown from the test results that the finer the filler particles, the more effective is the filler. This can readily be seen because (a) the smaller the particle size the greater is its surface-area which provides greater area for adsorption that leads to consistency change (b) the finer the filler particles the greater is their capability of remaining in suspension in the binder. Though the specific gravity of commonly used fillers does not vary to a great extent, it does affect the amount in suspension and it would be more important when new filler material is encountered.

10. In relation to the desirable fineness of the filler, the terminology colloidal suspension was suggested. According to the study, particle size of 10μ appears to be the dividing line of colloidal size. It should be noted that the amount in colloidal suspension, being dependent on many factors, cannot be related to the particle size of filler alone. However, from the practical point of view, the # 325 sieve can be used as a convenient yardstick in evaluating as well as specifying mineral fillers used in asphaltic paving mixtures.

11. In relation to specific gravity of the filler, two points of practical importance should be brought up:

(a) Other properties being equal, the use of low specific gravity filler is more desirable than a high specific gravity filler.

(b) In specifying the proportion of filler to be used in an asphaltic paving mix, either the amount should be expressed in volume (in addition to fineness) or the quantity in weight should be adjusted according to the specific gravity of the kind of filler to be used.

This investigation was carried out with limited number of types of filler and sizes of filler particle. It is believed that future work along this line with more filler types and particle sizes will reveal more information on the properties of asphalt cement mastics in relation to not only filler particle size and gradation but also filler particle shape and mineral composition.

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APPENDIX

Voids in Filler by Kerosene Absorption

Twenty grams of the dry, cool powder are placed in a five inch hemispherical iron dish. From a 25ml burette one to three drops of kerosene are added to the powder in the center of the dish. This forms a wetted ball. The dish is then shaken with a rotary motion so that the ball takes up all the powder that will adhere to it. Successively add a few drops of kerosene to the ball and shake until the wetted ball picks up all the dry powder. The end point is taken as the point at which the ball in being rotated first marks the inside of the iron dish.

The "kerosene number" of the filler is recorded as the milliliters of kerosene absorbed by 100 grams of the filler. This volume is of course five times the volume used to satisfy the 20 grams sample. Three tests should be made and averaged for each filler.

The "kerosene voids" are easily calculated from the "kerosene number" and specific gravity of the powder.

$$\text{Percent voids} = \frac{\text{Volume of liquid}}{\text{volume of liquid} + \text{solid volume of powder}} \times 100$$

$$= \frac{\text{kerosene number}}{\text{kerosene number} + \dfrac{100}{\text{sp. gr. of powder}}} \times 100$$

Determination of Bulk Density of Filler in Benzene

The determination is carried out in the following way. The filler is first dried in an oven at a temperature of 105-115 °C for fours hours and cooled in a desicator to room temperature. Ten grams of the filler is then weighed out into a boiling tube, about 150 mm long and 24 mm internal diameter, calibrated in c. c. The tube is nearly filled with pure, dry benzene and placed in anwater bath and the benzene is boiled for two minutes while the filler is stirred with a metal rod to remove trapped air. The tube is then allowed to cool to room temperature, after which the contents are thoroughly stirred with the metal rod so as to bring all the powder into suspension, and then allowed to settle undisturbed for at least six hours. The bulk volume of the filler (V) is then read off directly in c. c. and the bulk density evaluated from the equation

$$\text{Bulk density} = \frac{10}{V} \text{ g/c.c.}$$