# AGGREGATE SURFACE REACTION TEST TO EVALUATE STRIPPING IN BITUMINOUS MIXTURES

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# AGGREGATE SURFACE REACTION TEST
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# CHAPTER I

# INTRODUCTION

## Statement of Problem

Asphalt pavements comprise a mixture of asphalt material and mineral aggregate. Low traffic roads may be surfaced with a seal coat, consisting of a layer of asphalt material over which a layer of mineral aggregate is placed and rolled to imbed the aggregate particle in the asphalt. This type of pavement surface provides a watertight membrane over the road bed, and a relatively smooth surface which is free of dust and mud. Wheel loads are distributed by the base course material to the underlying subgrade.

More heavily traveled roads may be surfaced with asphalt concrete. This is a mixture of asphalt cement and mineral aggregate that is proportioned and mixed together at a relatively high temperature of approximately 300 F. The mixture is designed to provide a dense and stable surface layer when it is properly placed and compacted. The pavement surface layer helps to support the imposed wheel loads but its primary job is to transmit these loads to the underlying base and subgrade layers. Asphalt concrete pavement also provides a smooth durable riding surface which is impermeable by water.

The performance of the road or pavement, that is how well the traveling public can move safely over the road, has been a matter of opinion for some time. There are honest differences of opinion even among experts making subjective evaluations of just about anything. However, the serviceability of any highway may be expressed by the mean evaluation given to it by all people using the highway.

A method of evaluating pavement performance using a visual condition survey was reported by Ford and Bissett (1)[1]. They correlated the condition survey results with pavement deflections obtained using a Benkelman beam. A quantitative evaluation procedure was developed for the AASHO Road Test at Ottawa, Illinois, and reported by Carey and Irick (2). They used a rating panel to establish a present serviceability index (PSI) of a wide range of selected pavements containing all the various types and degrees of pavement distress likely to influence the serviceability of highways. A slope measuring vehicle, the Chloe profilometer, was then used to measure the roughness of the selected pavements. The numerical values obtained from the Chloe profilometer were correlated with results obtained from the rating panel to determine the PSI of a pavement. Studies made at the AASHO Road Test have shown that about 95 per cent of the information about the serviceability of a pavement is contributed by the roughness of its surface profile (2).

The factors thought to influence overall pavement performance include: structural design of the roadway, bituminous mix design, mineral aggregate properties, asphalt material properties, construction techniques, amount and character of traffic, environment of the road, and maintenance. The problem area of this study will be limited to the relationship between mineral aggregate and asphalt materials.

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[1] The numbers in parentheses correspond to the listing of references in the Bibliography.

In the United States there are approximately 755,000 miles of paved primary and secondary roads as reported in Highway Statistics (3). Of these, 82 per cent or 605,000 miles are bituminous surfaced roads. Many different types of aggregate and asphalt cement are used, depending upon local availability and experience. Supplies of high quality aggregates are diminishing as quarries and pits with a proven service record are depleted. New sources of aggregate are needed for major highway construction and reconstruction as is evidenced by the increased utilization of synthetic or manufactured aggregates. As continuing urbanization reduces the number of potential aggregate sources; re-evaluation and upgrading of aggregate previously thought unsuitable for use in bituminous mixtures may be desirable.

One area of asphalt pavement performance that has been a continuing problem for the highway engineer is pavement failure caused by stripping of asphalt cement from the aggregate in the mixture. Stripping occurs where there is loss of adhesion between the aggregate and the asphalt cement due primarily to the action of water. The resulting deterioration can be a serious problem and in some cases results in substantial reduction of total pavement performance. The Highway Research Board, in January, 1971, announced highway research project solicitations in the category of aggregates used in bituminous mixtures. They placed the subject, "Stripping of Asphalt Cements in Bituminous Mixtures", as the highest priority problem of 52 research studies that were desirable.

Factors which affect stripping include: aggregate properties, such as absorption, surface texture, porosity and mineral composition; and asphalt cement characteristics, such as surface tension, chemical composition, and viscosity. Thus, adhesivity of asphalt to aggregate is related to the physical and chemical properties of both the asphalt and the aggregate. This adherence is detrimentally affected by the presence of water, i.e., the bond at the solid-liquid interface is or can be disturbed and deteriorated by water action.

In general, siliceous aggregates have been classified as hydrophilic (water lovers) and tend to strip more readily than limestone aggregates which have been classified as hydrophobic (water haters). Hubbard (4) applied the term hydrophobic to an aggregate which will persistently retain an asphalt film in the presence of water. Practical experience reported by Mathews (5) indicates that relatively few aggregates are known which are completely resistant to the action of water under all conditions of practical use. Further, Mathews stated:

> For this reason it is not possible to generalize about the behaviour of classes of aggregates. In particular, the well-known and often quoted rule that 'acidic' rocks (high silica content) are vulnerable to stripping whereas 'basic' rocks (low silica content) are not, is quite wrong.

Perhaps, a better method of classification of aggregates was proposed by Mertens and Wright (6). The fact that both limestone and siliceous types of aggregate are readily wetted by water indicate that both types are truly hydrophilic in character. The terms proposed by Mertens and Wright are "electropositive" for limestone aggregates and "electronegative" for siliceous types of aggregates. These two types of aggregates represent the extremes found in aggregate classifications. Most aggregates contain elements of both electropositive and electronegative materials on their surfaces. A schematic classification system for aggregate, based on their system is shown in Figure 1.

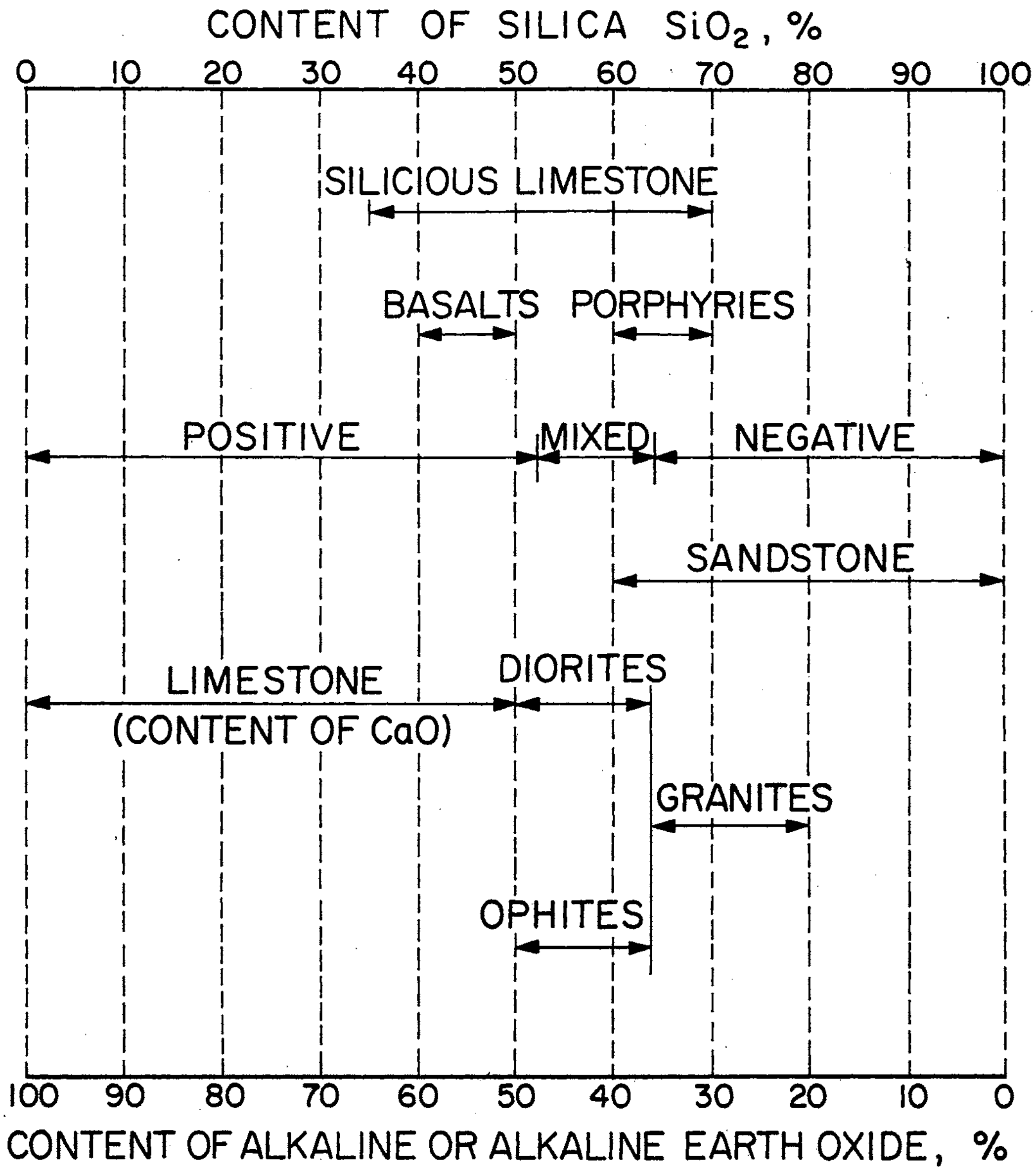

Figure 1. Classification System of Aggregate (After Mertens and Wright, 6)

In many areas the use of siliceous aggregates in paving mixtures has been curtailed because of bad pavement performance experiences. Frequently the surface of some asphalt concrete pavements are made with some limestone aggregates which tend to polish under traffic. This causes a reduction in skid resistance of the pavement surface. The need for improving or retaining skid resistance in surface mixtures has created interest in incorporating the harder less easily polished aggregates having a high siliceous content into the paving mixtures.

Presently, there is no standard test to quantitatively evaluate directly the amount of stripping that occurs when asphalt cement coated aggregates are subjected to the detrimental action of water. There is a definite need to develop a small-scale laboratory testing procedure which will measure stripping in more quantitative terms.

Previous investigators have attempted to determine the effects of water on the adhesion between the aggregate and the asphalt cement. Since 1932, tests on compacted bituminous mixtures have included: immersion compression, cold water abrasion, sonic vibration, water susceptibility, and laboratory test tracks. Tests proposed on asphalt cement coated aggregate include: static immersion, dynamic immersion, chemical immersion, and stripping coefficient. Methods investigated to measure in quantitative terms the amount of stripping of asphalt cement coated aggregate include: radio isotope tracer, light reflection, mechanical integration, tracer salt, and dye adsorption. Despite the large number of tests devised to study the effect of water on adhesion, an examination of the technical literature reveals a continuing effort of research studies being conducted to evaluate stripping (7).

## Method of Study

Previous tests were considered either too complex, requiring extensive equipment and expertise, or their accuracy was questionable since results were based on the operator's visual judgement of the amount of stripping. The general procedure proposed for this study was to measure the surface area of an aggregate sample before mixing with asphalt cement, and then using a similiar coated sample to measure the surface area stripped of asphalt cement after exposure to water action. The problem was to obtain a quantitative measure of the aggregate surface area which had been stripped of asphalt cement after suitable exposure to the action of water. It was reasoned that if the stripped aggregate surface was exposed to a suitable reagent, e.g., an acid which would not react with the asphalt, the resulting reaction between the acid and aggregate would liberate a gas as well as a certain amount of heat. The amount of these reaction products should be proportional to the amount of exposed aggregate surface area.

For example, when hydrochloric acid (HCl) is added to limestone, which is predominantly calcium carbonate ($CaCO_3$), the following reaction occurs:

$$CaCO_3 + 2HCl \longrightarrow CaCl_2 + H_2O + CO_2\uparrow + \text{heat}$$

If the aggregate is predominantly siliceous ($SiO_2$), and hydrofluoric acid (HF) is added, the following reaction occurs:

$$SiO_2 + 4HF \longrightarrow 2H_2O + SiF_4\uparrow + \text{heat}$$

For aggregates of mixed composition, suitable proportions of HCl and HF may be combined to obtain a surface reaction.

## Purpose and Scope of Study

The purpose of this study was to develop a laboratory test device to measure the gas pressure and heat liberated when a suitable acid was added to an aggregate sample. A second laboratory device was developed for the purpose of subjecting asphalt cement coated aggregate to water effects and expediting the stripping process. One high quality asphalt cement (85-100 penetration) was used in all tests.

Eleven selected aggregates, used in Oklahoma highway construction, were evaluated for their relative stripping tendency by the surface reaction test. These aggregates included: limestone, siliceous limestone, chert, chert gravels, siliceous gravels, and sandstones. Test results were correlated with results of the ASTM "Test for Coating and Stripping of Bitumen-Aggregate Mixtures" (8) obtained at 140 F, and results of the ASTM "Test for Effect of Water on Cohesion of Compacted Bituminous Mixtures" (9) obtained using a modified test procedure employing the Marshall stability testing head.

# CHAPTER II

## REVIEW OF LITERATURE

The phenomenon of stripping has been studied by many investigators during the past 40 years. By the end of 1955 over 400 papers and reports on adhesion in bituminous materials had been published (5). A 1968 report, "State of the Art: Effect of Water on Bitumen-Aggregate Mixtures", by Majidzadeh and Brovold (7) contains reference to 265 papers and reports.

Much of this research has been directed toward determining the cause or causes or stripping with emphasis on relating laboratory test results to observed pavement performance. Other research emphasis has been placed on improving the stripping resistance of a particular mixture by additives and evaluating minimum quantities of additives required to achieve a satisfactory pavement mixture.

The magnitude and distribution of stripping problems may be emphasized by analysis of the results of a 1952 poll (10). A questionaire on adhesion tests was submitted to 51 agencies (including 48 state highway departments). Of the 40 replies obtained 52 per cent felt that stripping was a problem in their area, however, only 40 per cent used an adhesion test. A 1958 report showed that 21 state highway departments had coating and stripping test requirements (11). A majority of these test procedures will be discussed later.

## Stripping Phenomenon

Field observations of stripping indicate different modes of stripping occur. It may progress from the bottom of the pavement upward or from the top downward. Studies by Pauls and Goode (12) on hot-mix hot-laid pavements indicated that stripping began at the bottom of the pavement layer and slowly progressed upward. In another study, the rate of progress was thought to be related to the density of the pavement (13). In most cases, damage was not apparent until after the pavement had failed over a large area (14).

In the discussion to this paper by Pauls and Goode (12), Mr. A. W. Dow stated:

> I made a report to the Engineer Commissioner . . . on the 'Failure of Asphalt Pavements and Their Causes' . . . This [pavement] disintegration manifests itself differently, depending on the character of the pavement. If the asphalt surface is soft or the concrete [base] smooth, the first defect noticed will be the tendency of the pavement to crowd in warm weather. This is due to the under portion of the surface mixture rotting, so to speak, thus destroying the cementing properties of the asphalt. The upper portion although good, being deprived of the support of the affected mixture under it, will be crowded out by traffic . . .
>
> In cases where the concrete base is rough and the surface mixture hard, the principal disintegration will take place in cold weather, nothing abnormal being noticed until the pavement begins a rapid crumbling away in the affected spot under traffic. On examining a section of asphalt surface disintegrating from this cause, especially where it has not been going on for too long a time, there will be found a layer of perfectly sound and good material at the surface of the pavement while underneath the mixture will show evidence of being disintegrated by water--that is, the sand will appear clean and white in spots as though there had been an insufficiency of asphalt cement to cover it. The concrete base under the affected pavement will generally be found damp or even wet.

In another study (15) a road-mix bituminous surface, composed of slow-curing liquid asphalt and local pit run aggregate, suffered rapid deterioration of the surfacing soon after construction when the first rains fell. The surface softened to a slight depth after each subsequent rain, and became slimy under traffic. Upon drying, the surface raveled leaving the larger aggregate particles exposed.

Parr (16) cites several examples of unsatisfactory behavior of bituminous surfaces under traffic as a result of exposure to water after construction. Several conditions which can render a hot-mix bituminous pavement susceptible to absorption of water and subsequent failure to stripping are: 1) high voids in mixture, 2) poor aggregate gradation, 3) insufficient asphalt cement, 4) inadequate compaction, and 5) adsorption of asphalt cement by clay fines.

Parr also found that moisture contributes to poor performances, i.e., stripping, of seal coat applications under the following circumstances: 1) retardation of curing due to high humidity, 2) excessively wet cover aggregate at time of construction, 3) presence of dust or clay fines in the cover aggregate, 4) inadequate or nonuniform asphalt application, 5) excessive amount and speed of traffic during curing period, 6) inadequate compaction of cover aggregate, and 7) adverse weather conditions at time of or immediately following construction.

Hubbard (4) discussed the important factors that must be considered to ensure pavements of maximum durability against water action. Briefly, these included: thorough wetting of the aggregate with asphalt is essential to good adhesion; use an asphalt having the highest practical viscosity for the specific application, as this is an important deterrent to stripping; time is often an important element in developing the maximum adhesion; and avoid the use of highly hydrophilic aggregates, as they may lose their adhesion to asphalt films in the presence of water.

Thus, it is apparent that stripping is a complex phenomenon influenced by many variables related to the materials involved, ambient conditions of weather and loading, as well as to construction practices and roadway design techniques. These all affect the adhesivity between the aggregate and binder used.

In order for stripping to occur there must be a loss of adhesion between the asphalt binder and the aggregate particles used in a paving mixture. Thus a great deal of research effort has been devoted to the study of the adhesion characteristics that exist between these two materials. A number of theories on adhesion and mechanisms of stripping have been developed by various investigators.

## Theories of Adhesion

Adhesion is defined as that physical property or molecular force by which one body sticks to another of a different nature (17). Factors which affect the adhesion of asphalt to aggregate include: surface tensions of asphalt and aggregate, chemical composition of these materials, viscosity of the asphalt, surface texture of the aggregate, porosity of the aggregate, cleanliness of the aggregate, and dryness and temperature of the aggregate when mixed with asphalt (18). Four major theories which have been formulated as to the cause of adhesion are: chemical reaction, mechanical adhesion, surface energy, and molecular orientation (7).

Chemical Reaction. This theory is credited to Riedel (19) and states that the acidic components of the bituminous material react with basic minerals of the aggregate to form water-insoluble compounds at the interface. While this theory may have some basis in fact, it does not hold true in all cases since good adhesion has been reported between siliceous aggregate and some asphalts by Winterkorn et al. (20). It is generally believed that siliceous aggregates, which are acidic, tend to strip more readily than basic aggregates, such as limestones (4, 6, 21).

<u>Mechanical Adhesion</u>. The aggregate properties considered to affect mechanical adhesion are: surface texture, absorption and porosity, surface coatings and area, and particle size. Lee (22) observed that rough, irregular surfaced aggregate had better retention of asphalt than smooth glassy surfaced aggregate. Once wetted, a rough textured surface will produce greater adhesion under service conditions than a smooth surface.

It has been shown that different aggregates adsorb asphalt to different degrees (5). Some components of asphalt, primarily the oily constituents, enter the pores or capillaries of an aggregate particle where they are preferentially adsorbed. This causes the asphalt coating on the particle to become harder. The interlock of the asphalt coating with these pores should make the asphalt adhere more strongly and be less readily stripped by water (18). However, Tyler (23) found no correlation between absorptive capacity of an aggregate and the amount of stripping resistance.

Nicholson (17) presented test results showing that colloid coatings on sand grains incorporated in a bituminous mixture tend to reduce the mixtures resistance to stripping. Thelen (18) observed that dust on aggregate surfaces had a tendency to trap air when treated with road oils or cutback asphalts and weakened the bond by preventing intimate contact between the aggregate and the asphalt. Thus, stripping is promoted by providing channels at the interface where water can penetrate.

An investigation by Stross and Anderson (24) reported the importance of the particle size on adhesion in bituminous mixtures, particularly material sizes below the No. 200 sieve. Their test results showed that use of aggregate containing appreciable amounts of clay required far more asphalt for complete coating than was compatible for mechanical stability. Thus, a stable mixture, with excess fines, would tend to strip because of the presence of partially uncoated aggregate particles.

Surface Energy. The ability of asphalt to make intimate contact with the surface of the aggregate is known as its wetting power. The wettability of the solid surface of the aggregate is its ability to be covered by the asphalt (4). The wetting power of an asphalt is largely controlled by its viscosity. Viscosity is that property of a liquid that represents a resistance to flow caused by molecular friction. Viscosity is also related to surface tension, which is the force tending to hold a drop of it in spherical form. Water has a higher surface tension than most liquid asphaltic materials but its lower viscosity makes it a better wetting agent.

When asphalt spreads over and wets the aggregate surface a change in energy takes place which is called adhesion tension (25). This adhesion tension is a surface phenomenon and depends upon closeness of contact, mutual affinity of the two materials and time of contact. Numerically, adhesion tension is equal to the sum of the surface tensions of the asphalt and aggregate less the interfacial tension between them.

Results of measurement of the surface tension, interfacial tension and adhesion tension of the ingredients of some bituminous mixtures were presented by Rice (25), and are shown in Table I. McLeod (26) noted that an aggregate tends to become coated by the liquid present for which

## TABLE I

### VALUES FOR SURFACE, INTERFACIAL AND ADHESION TENSION
(After Rice, 25)

| Property | Value (ergs/cm$^2$) |
|---|---|
| Surface Tension | |
| Water | 72 |
| Asphalt | 26 to 39 |
| Various marbles of limestone | 28 to 50 |
| Diabase | 42 to 50 |
| Various granites | 52 to 73 |
| Interfacial Tension | |
| Asphalt-water | 25 to 35 |
| Quartz-water[1] | 0 |
| Quartz-asphalt | 14 to 20 |
| Glass-tar | 18 |
| Adhesion Tension | |
| Limestone-asphalt | 21 to 26 |
| Slag-asphalt | 23 to 26 |
| Sand-asphalt | 22 to 30 |
| Sand-tar | 40 |
| Quartz-asphalt | 75 |
| Limestone-water | 58 to 64 |
| Slag-water | 63 to 68 |
| Silica sand-water | 83 |

[1] Based on the assumption that the surface consists of a water film.

it has the greatest adhesion tension. Results of tests performed by Mack (27) on energy relations at the interface between asphalt and mineral aggregate indicate that the interfacial tensions vary not only with the type of aggregate but also with the type of asphalt. Based on the data presented in Table I, Rice observed that in all cases, except for the quartz-asphalt interface, the adhesion tension for water to aggregate was higher than for asphalt to aggregate. Thus, according to the surface energy theory, water will tend to displace asphalt at an interface where contact is made between water-asphalt-aggregate.

Molecular Orientation. According to Hubbard (4), when molecules of asphalt come in contact with the aggregate surface they orient themselves so as to satisfy all energy demands of the aggregate to the best of their ability. This orientation in viscous liquids and semisolids proceeds rather slowly, and considerable time may elapse before the maximum adhesion between an asphalt film and an aggregate surface is developed. Water molecules are all dipoles, whereas asphalt molecules predominately appear to possess non-polar characteristics, so that water possesses an advantage over asphalt in rapidly satisfying energy demands of polar aggregate surfaces. However, asphalt dipoles may have a more powerful energy demand for some aggregates than do water molecules, and be able to displace water from the surfaces of these aggregates.

## Mechanisms of Stripping

An asphalt mixture is a system composed of asphalt cement, aggregate and air. Water may be present due to incomplete drying of the aggregate or may be derived from external sources after construction. Stripping in this system occurs when the bond between the aggregate and asphalt is broken by water. Stripping is a reverse process to that of adhesion. Water through some mechanism causes the bond between aggregate and asphalt to be diminished. Four mechanisms of stripping that have been advanced include: detachment, displacement, film rupture, and hydraulic scouring (7, 28).

Detachment. This is the case where the asphalt cement is separated from the aggregate surface by a thin film of water but there is no obvious break in the continuity of the asphalt coating. The water may be present in the capillary pores of the aggregate due to improper drying, or may be due to the diffusion of water through the asphalt layer. In this state, the asphalt film can be peeled cleanly away from the aggregate. Hughes, Lamb and Pordes (21) suggested that the crystal lattice of the mineral reacted with water to form a gel-like structure and detachment was partially due to the rupture of this weakened structure.

Displacement. Mathews (5) stated that it was unusual for the bond between a binder and an aggregate to fail at the interface for reasons other than the displacement of the binder by water. Lee (22) performed experiments to measure the equilibrium forces which act on the binder-water-aggregate system. He attributed the displacement of binder to the superior wetting properties of water.

The surface energy necessary to strip asphalt from aggregate was reported by Thelen (18). His work is repeated here as a numerical example. Normal stripping is illustrated in Figure 2. This is where a discontinuity or break in the asphalt coating occurs and asphalt, free water and aggregate are all in contact. The free energy change is:

$$\Delta F = \gamma_{12} + \gamma_{24} - \gamma_{14}$$

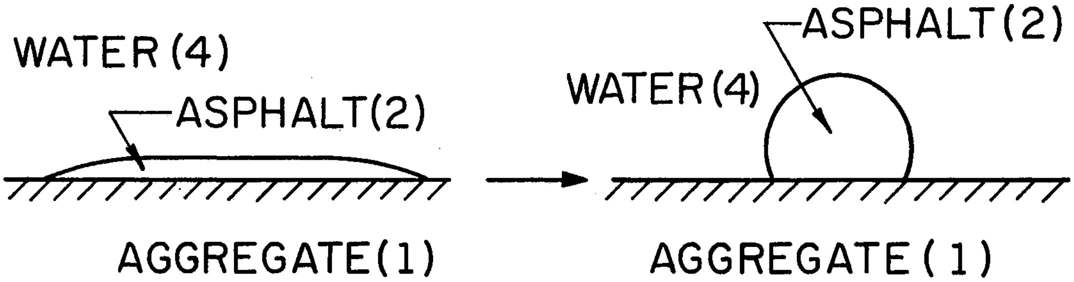

Figure 2. Normal Stripping (After Thelen, 18)

where: $\Delta F$ = Free Energy Change

$\gamma_{12}$ = interfacial tension, aggregate-asphalt

$\gamma_{24}$ = interfacial tension, water-asphalt

$\gamma_{14}$ = interfacial tension, aggregate-water

from data of Table I,

$$\gamma_{12} = 17 \text{ ergs/cm}^2$$

$$\gamma_{24} = 30 \text{ ergs/cm}^2$$

$$\gamma_{14} = 0 \text{ ergs/cm}^2$$

then

$$\Delta F = 17 + 30 - 0 = 47 \text{ ergs/cm}^2$$

which is the energy potential to cause stripping. The rate at which the displacement occurred depended on the magnitude of the free energy evolved, and the viscosity of the asphalt. High viscosity binders show higher resistance to displacement; thus road surfaces that are subjected to rain before they attain their desired viscosities may show a higher degree of stripping (5). Displacement fails to explain how stripping is initiated when the aggregate is completely coated with asphalt.

<u>Film Rupture</u>. This method of stripping may occur when adhesion of the asphalt cement is not uniform over the entire surface of the aggregate (4). It has been suggested by Mathews (5) that the asphalt film was thinnest at sharp corners and edges of the aggregate and the effect of traffic may cause the film to fracture, thus initiating stripping. Once ruptured, and in the presence of water, the asphalt film would tend to take up the form of lowest potential energy by retracting to spherical globules (21).

<u>Hydraulic Scouring</u>. This theory on the mechanism of stripping was presented by Stevens (28). He stated that voids in a bituminous mix or

seal-coat consist of a largely interconnected pore system which is partially filled with air and water. In a saturated pavement on impact of a wheel, water is pressed into the pavement in front of the tire and sucked out as the tire leaves the spot; rapid decompression within the pore system takes place, and a small upward thrust is developed. This movement of water, due to the action of traffic, would tend to strip hydrophilic aggregate spontaneously, and dust may become mixed with rainwater and assist in abrading the asphalt-aggregate contacts. This theory may explain failure of pavements in the field due to action of water and traffic.

### Summary

No one theory of adhesion or mechanics of stripping can explain the phenomenon of stripping. The resulting physico-chemical forces acting at the interface between asphalt cement, aggregate, water and air may promote stripping. The displacement and detachment mechanisms may be considered the primary causes of stripping, while film rupture and hydraulic scouring are secondary causes. Adhesion of asphalt cement to the aggregate seems to be controlled by the characteristics of the aggregate, with the asphalt cement characteristics being secondary.

## Tests for Stripping

Asphalt-aggregate systems are divided into two types, two dimensional and three dimensional, by Majidzadeh and Brovold (7). The two-dimension binder-aggregate system is comprised of cases in which the asphalt and uniform sized aggregate form a layered and laminated structure. These may be thought of as surface treatments or seal coats.

In the second type of system large numbers of well graded aggregate particles are held together in a coherent three-dimensional honey comb structure by the asphalt cement. This asphalt-aggregate system comprise hot-mix asphalt concrete or road mix type mixtures.

Stripping tests will be discussed based on whether the bituminous mixture is considered two-dimensional (layered system) or three-dimensional (compacted bituminous mixture).

## Layered System of Construction

Tests devised for this system have a common procedure. The aggregate to be evaluated is usually of one gradation, commonly sized to pass a 3/8 in. sieve and be retained on a 1/4 in. or No. 4 sieve. The aggregate is coated with the asphalt material, subjected to the effects of distilled water, and then evaluated to ascertain the percentage of asphalt coating still adhering to the aggregate.

Several different detailed procedures have been proposed. Four types reviewed here are: dynamic immersion stripping test, static immersion stripping test, chemical or boil test and detachment test. The laboratory test track, which will be discussed with the compacted bituminous test, has also been used to evaluate stripping tendencies of the layered system of construction.

Dynamic Immersion Stripping Test. Nicholson (17) devised a dynamic immersion stripping (DIS) test to evaluate the adhesion of asphalt to various types of fine aggregates. His test mixtures represented sheet asphalt and typically were composed of: 12 per cent limestone dust (passing No. 200 sieve), coarse concrete sand and 8 per cent asphalt. He weighed out 50 g of the cooled friable mix and placed it in a 250 ml

Pyrex Erlenmeyer flask. About 175 ml of warm (140 F) distilled water was added to the flask before placing it in a shaking machine. The machine was rotated at 39 rpm submerged in a water bath whose temperature was held at 140 F during the one hour test period. The sample was then removed from the machine and the contents visually examined to see if the asphalt had washed off the aggregate.

Dow (29) used the same type test to evaluate his Colprovia Paving Mixture in 1936, except his mixtures were shaken at intervals of 1, 3, 5, 10, 15, and 30 minutes. The mixture was evaluated at the end of each interval and any mixture which did not strip at the end of the 30 minute period was passed as satisfactory. Dow ran tests on various component parts of the aggregate used in his mixture to determine which aggregate size contributed to stripping. Winterkorn, et al. (20) used a similar test procedure but varied the test temperature and rate of rotation, and extended the time of rotation up to 4 hours in length.

Lang and Thomas (30) also used the DIS test devised by Nicholson. Their report gives the results of tests on 6 different types of aggregate using 23 different brands of asphalt cement (85-100 penetration grade). The samples were rotated at 44 rpm for two 15 minute periods at 75 F, and for additional 15 minute periods at 100 F and 120 F. At the end of each 15 minute period the samples were examined and the visually estimated percentage of surface area of the aggregate which had been stripped was recorded. Two of the same aggregate types were also subjected to stripping after coating with a 50-60 penetration grade asphalt cement. Considerable variation in the stripping tendency of different asphalt cements were noted. Average results of these stripping tests are shown in Table II.

TABLE II

DYNAMIC STRIPPING TEST RESULTS
(After Lang and Thomas, 30)

| Aggregate | Per Cent Coating | |
|---|---|---|
| | Penetration Asphalt Cement | |
| | 50-60 | 85-100 |
| Granite | -- | 34 |
| Quartzite | 75 | 53 |
| Feldsite | -- | 65 |
| Traprock | -- | 91 |
| Limestone | 99 | 95 |
| Mixed Gravel[1] | -- | 99 |

[1] Mixed gravel composed of 32% limestone, 39% granite, 27% traprock, 1% sandstone, and 1% quartzite.

Critz and Goode (31) used a DIS test to evaluate the effect of using additives to decrease the stripping of bituminous coated aggregate. They concluded that the conditions of the test were not sufficiently severe to demonstrate differences between the additives or the effect of using different percentages of additive.

A modification of the Nicholson DIS test was used by Tyler (23) to increase the stripping effect on the bituminous coated aggregate. In this method the coated aggregate, after curing, was separated by hand and immersed in a mason fruit jar containing 1000 ml of distilled water. The jar was then placed in a Ro-Tap sieve-shaker and agitated for 30 minutes. The samples were removed from the jar and graded according to visual inspection and count. Tyler concluded that aggregate composed of granite, quartz and some cherts were hydrophilic in nature and aggregate types such as dolomite, traprock, limestone and basalt were hydrophobic.

Sanderson (13) used the Tyler test in evaluating the stripping resistance of seven different aggregates. These aggregates were also treated with methylchlorosilanes to determine if their stripping resistance would be increased. Sanderson concluded that the Tyler test did not simulate actual road conditions. Since the particles of aggregate were agitated violently in a jar of water, it seemed to him that most of the stripping which occurred was caused by mutual abrasion of the aggregate particles.

The State of California Highway Department laboratory test manual describes a dynamic immersion method of test to evaluate film stripping (32). The test is applied to the aggregate fraction passing the 3/8 in. sieve and retained on the No. 8 sieve. After curing, the coated sample is immersed in water at 77 F and agitated for 15 minutes. Additional 15 minute periods of agitation successively at 100 F and 120 F are used in special cases.

<u>Static Immersion</u>. In 1939, Hubbard (4) suggested a test to evaluate resistance to film stripping. The procedure was to prepare laboratory samples with the same proportions of asphalt and aggregate as that intended for the mixtures in the field. After mixing was completed, the blended materials were spread out in a thin layer and allowed to stand for 24 hours. A suitable amount of the cold mixture was then placed in a glass jar with a screw top lid, and completely covered with distilled water. The jar and its contents remained undisturbed for 24 hours after which the mixture was examined for evidence

of film stripping. The jar was then vigorously hand shaken for three periods of 5 minutes and the mixture examined at the end of each period. If only a slight amount of stripping was noted at the end of the third period of shaking, little or no trouble was anticipated with stripping under ordinary field conditions. Results of test by Lee (22) using a jar test similiar to the one described by Hubbard, were published in 1936.

A water-asphalt preferential test intended to determine the water-resisting properties of mineral fillers used in a bituminous mixture was described by Stanton and Hveem (33). This test consisted of mechanically mixing 50 ml of a heavy fuel oil heated to 140 F with 10 g of filler dust (passing the No. 200 sieve) for 5 minutes. Then 100 ml of distilled water at 140 F was added and the mixture stirred 5 additional minutes. The jar was then set aside, allowing the sample to settle until the water became clear. The amount of clean filler in the bottom of the jar was estimated and recorded as the percentage of total filler. Higher percentages of a poor quality filler separated from the oil and collected at the bottom of the jar.

An evaluation of stripping test methods and their usefulness was published by Holmes in 1939 (34). He divided the various tests into three categories as: partition tests, displacement tests and abrasion-displacement tests. The partition test of Holmes was similar to the water-asphalt preferential test previously described by Stanton and Hveem, while the abrasion-displacement test was similar to the dynamic immersion test where the coated aggregate was shaken in a horizontal plane. Holmes thought both of these tests were of little value to evaluate aggregate stripping tendency.

The displacement test was classified by Holmes as the one most frequently used at that time and was essentially the same test used by Hubbard and Lee, with slight modifications. The coating ability of the asphalt before immersion as well as the stripping resistance at the end of the test were noted. For the water-displacement test at 140 F, Holmes coated No. 4 to No. 10 sieve size material with 4 per cent cutback asphalt. The mixture was cured 2 days at 140 F, then completely covered with distilled water for 18 to 20 hours at 140 F. The specimen was then cooled to room temperature and spread out and dried. The percentage of aggregate still coated with asphalt was determined by visual estimation.

The influence of the pH of water on asphalt stripping from aggregate was reported by Gzemski (35). The static immersion test previously described by Hubbard was used in this study. The coated aggregate was cured 48 hours at 110 F and then immersed at 77 F in a water solution (whose pH was varied from 4 to 10) for 24 hours. The percentage of asphalt coating that remained on the aggregate at the end of the immersion period was determined by visual estimation while still under water. Low pH solutions favored the retention of asphalt on the hydrophilic aggregate (rhyolite and granitic gneiss), while with dolomite better retention was obtained at pH values of 8 to 10. The pH of natural waters was surveyed to determine the extent of the variation of this factor. In Pennsylvania during 1944-45, the pH of surface waters varied between 2 and 10. The majority of these pH values were in the range of 4.5 to 8.5.

Carroll (36) reports that rainwater in equilibrium with the $CO_2$ of the atmosphere (as in clouds) has a pH of 5.7, and in the United States the pH of rainwater is generally between 6 and 7. According to Carroll, all water that comes in contact with rocks is slightly acid. When mineral grains are crushed and placed in water, the pH obtained is known as the "abrasion pH." Carroll reports abrasion pH values for common minerals as: quartz, pH 6-7; feldspar, pH 8-9; amphiboles, pH 10-11; pyroxene, pH 8-10; mica, pH 7-9; calcite and dolomite, pH 8-10; and clay minerals, pH 6-7. The test results of Gzemski perhaps reflect the tendency of a bituminous material to adhere better in the presence of a water whose pH is compatible with the abrasion pH of the aggregate to which it is applied.

A report of ASTM Subcommittee B-26 on the effect of water on bituminous coated aggregate was published in 1952 (37). A survey of existing methods was used as a basis for proposing a standard method of performing the static immersion stripping (SIS) test. This standard method of test was adopted by ASTM, after continued review, in 1959 (8).

The ASTM procedure is applicable to cut-back, emulsified, and semi-solid asphalts and tars. The method consists of coating 100 g of pre-washed, selected aggregate (passing the 3/8 in. sieve and retained on the 1/4 in. sieve) with 5.5 per cent of the liquid or semi-solid bitumen, or 8 per cent of emulsified asphalt. Temperature of mixing is as required to obtain a satisfactory coating and curing conditions are compatible with the type of bituminous material employed. The coated aggregate is immersed for 16 to 24 hours in distilled water of 6.0 to 7.0 pH at room temperature (77 F). By observation through the water, from above, the percentage of the total visible area of the aggregate which remains coated (above or below 95 per cent) is estimated. This method should not be used as a field control measure because such correlation has not been established.

Karius and Dalton (38) evaluated the stripping tendencies of aggregate used for seal coats with a detachment test. The aggregate (5/8 in. to 1/4 in. in size) was placed on the prepared bituminous film under dry conditions. After curing 16 hours at 68 F, any loose aggregate was removed from the pan, and the weight of attached aggregate determined. The test pans were immersed in a water bath at 68 F and the detachment of the asphalt was observed daily for a period of 30 days. Percentage detachment was determined on a weight basis.

Boiling or Chemical Immersion. This type of test was originally developed by Riedel and Weber (39). The test involved placing a sample of bituminous coated aggregate in boiling water for 1 minute and noting if separation of the bituminous material occurred. The aggregate was classified as hydrophobic if there was no separation. The degree of adhesion was then determined by the resistance to stripping of the bituminous films when boiled for 1 minute in sodium carbonate solutions of increasing concentrations. A fresh sample of coated material was used with each solution. The numerical value of adhesiveness (on a scale of 1 to 10) was then defined by the concentration of sodium carbonate solution at which stripping occurred.

Winterkorn, et al. (20) compared results of the Riedel and Weber test method with those obtained using a dynamic immersion stripping test. They observed that both gave comparative results, but the dynamic immersion stripping test approached road conditions more closely than did the boiling test. Others have objected to the use of the boiling test because subjection of the sample to such high temperatures and exposure to the action of sodium carbonate solutions bear no relation to normal road conditions (5, 13).

Holmes (34) reported a water-boil test, where the coating of the aggregate was accomplished in the same manner as in his water-displacement test at 140 F. Then the coated aggregate was placed in a beaker and covered with distilled water. The beaker contents were heated to boiling in 6 minutes and boiled for 1 minute. The boiling sample was then cooled under running water and spread out on a flat surface. The amount of coated surface was estimated visually.

## Quantitative Methods To Determine Stripping

A problem, common to the previously enumerated stripping test procedures, has been the visual estimation of stripped surface area. Visual estimation provides only a qualitative indication of the stripping tendency of an asphalt-aggregate mixture and depends largely on the judgment of the operator, i.e., the individual performing the test. Considerable research effort has been expended to develop a more quantitative method to measure the amount of stripping that occurs. These procedures include the use of radioactive isotope tracer, lithium tracer-salt, dye adsorption, mechanical integration, leaching and stripping coefficient, for "quantity" measurements.

Radioactive Isotope Tracer. The aggregate to be evaluated was soaked in a radioactive calcium chloride solution and then dried. It was coated evenly with asphalt and the mixture was covered with water for a specified period of time. If the asphalt separates from the aggregate during immersion, the radioactive calcium on the surface of the aggregate dissolves in the water. After evaporation, the radioactivity of the residue was measured with a scintillation counter. A correlation between the radioactive count of uncoated and partially coated aggregate indicates the amount of stripping that has occurred (40). The radioactive tracer method has been criticized because the aggregate surface is changed by application of the calcium chloride. Also, this method has failed to gain general favor because of its hazard potential (7, 11).

Lithium Tracer-Salt. This method was developed by Brown, Sparks and Marsh (11). The aggregates were impregnated with a water-soluble lithium chloride salt, then oven dried and stored over water until ready to test. The aggregate used was sized to pass the No. 4 sieve and to be retained on the No. 6 sieve. A 90 gram test batch of aggregate was coated with 5.5 grams of RC-2 cutback asphalt. After mixing and curing the mixture was covered with 30 ml of distilled water in a screw cap jar and allowed to stand quiescent at 77 F for 20 hours. The percentage of retained coating was determined both visually and with the flame photometer.

The amount of lithium in the stripping water was determined with a flame photometer. By measuring the amount of lithium released to the water by an uncoated aggregate sample, the amount of exposed or stripped area was taken to be proportional to the change in concentration of lithium. It was noted that the flame photometer method of evaluation required additional instrumentation at appreciable cost. No mention was made as to what effect the lithium salt coating on the aggregate had on the adhesivity of the asphalt.

Dye Adsorption Method. Hveem (41) reported the work done on dye adsorption by the late A. R. Ebberts. Ebberts discovered that safranine dye was preferentially adsorbed by aggregate, but adsorbed slightly or not at all by asphalt. By proper adjustments of the original dye concentration, it was possible to measure the amount of adsorption, and relate it to exposed surface area.

This technique was revised and used by Skog and Zube (42) to evaluate stripping tendencies of the entire mix gradation for any source in which all aggregate surfaces have approximately equal dye adsorption. It was noted that mixture combinations that consisted of aggregate which had very low adsorption and high stripping and aggregate which had high adsorption and low stripping could provide misleading results.

Lortscher, Snyder and Filbert (43) reported a test method using fluorescein dye as a precoating material. The aggregate was coated with the dye, dried, coated with asphalt and subjected to a stripping test. As stripping occurred, the exposed dye dissolved in the stripping water. Evaluation of dye concentration was made with a colorimeter. By comparison of uncoated and partially coated dye concentrations the relative amount of stripping was determined.

Holmes (34) reported attempts to evaluate the exposed uncoated aggregate surface by means of adsorption of a standardized dye solution were unsuccessful because of the apparent tendency of the asphaltic material to often leave a thin invisible film of resinous material over the apparently exposed mineral surface, and because of adsorption of the dye itself by the asphalt.

Mechanical Integration Method. An apparatus was devised to assist the operator in systematically evaluating the amount of exposed aggregate surface after stripping. The apparatus consisted of a motor-driven stage that moved the sample relative to the operator's eye. The operator systematically observed the specimen through a low-powered microscope. When the edge of a non-coated area appeared the operator depressed a key to an electrical counting circuit, when the non-coated area had passed and asphalt appeared, he released the key. This counting process resulted in determining the percentage of stripped area. The large variation in results of visual estimates by different operators emphasized a very serious drawback to this method (43).

Leaching. This method of determining percentage stripping was reported by Lortscher, Snyder and Filbert (43). The method consisted of reacting hydrochloric acid with limestone aggregate and measuring the decrease in strength of the acid, as the chemical reaction took place. By reacting an uncoated aggregate specimen with acid, the rate of acid depletion observed was taken to be proportional to the aggregate surface area. A coated and partially stripped sample was then reacted with acid. The rate of acid depletion in this case was assumed proportional to the exposed or stripped surface area. The percentage of stripping was then calculated from the respective rates of acid depletion.

Stripping Coefficient. Thelen (18) proposed a test to evaluate the adhesivity of asphalt to aggregate in the presence of water. A representative aggregate particle was ground to a smooth surface and placed in a test cell. The aggregate temperature was raised, to approximate the hot-mix plant mixing temperature, in the presence of nitrogen or deaired air. A drop of molten asphalt was dropped on the solid aggregate surface and allowed to wet the surface. Heating was stopped and the temperature dropped to about 176 F. The cell was filled with distilled water at 176 F and held one hour. The silhouette of the asphalt film was then projected onto sensitized paper and its contact angle to the aggregate measured. The stripping coefficient was then calculated by the following equation:

$$\text{Stripping Potential} = \gamma_{24}(1 - \cos \theta)$$

where: $\gamma_{24}$ = interfacial tension between asphalt and water

$\theta$ = asphalt-aggregate contact angle

Thelen reported the interfacial tension of asphalt-water ranged from 25 to 35 ergs per square centimeter. No stripping coefficient values for aggregate was reported.

## Compacted Bituminous Mixtures

Tests which have been used to evaluate the water resisting characteristics of the entire compacted bituminous mixture include: immersion-compression (I-C), laboratory test tracks, vertical swell, water susceptibility, abrasion weight loss, and sonic vibration. Each test provides some measure of the change in a physical property of the mix due to the effects of water. This change in physical property is then related to stripping effects of water on the bituminous mixture.

Advantages attributed to tests of compacted bituminous mixtures over stripping tests of coated aggregate particles, include: 1) the test results are quantitative values, 2) the compacted test specimens represent the actual bituminous mixture that will be used in highway construction, and 3) the laboratory specimens are subjected to water action which may simulate actual field conditions, where the pavement mass is exposed to effects of rain and ground water. These advantages are offset somewhat by the necessity for more elaborate test equipment and test procedures that are required to achieve satisfactory test results.

Immersion-Compression Tests. Developmental work on this method was reported in 1943 by Krchma and Loomis (44). They compacted their mixtures using a vibratory procedure. The 3 in. diameter by 2 in. high specimens were cured 16 hours at 77 F before being tested in unconfined compression to determine their initial or dry strengths. Duplicated specimens were allowed to stand in water at 77 F and tested in compression after varying periods of time. Three different types of aggregates were evaluated using a 142 penetration Wyoming asphalt cement. Asphalt coated aggregate samples were also subjected to the dynamic stripping, boil, and swell tests.

The unexpected failure in 1941 of an experimental highway built in Colorado was attributed to the stripping effects of water. This failure prompted research on stripping by the Public Roads Administration. Pauls and Rex (15) reported the work undertaken by that agency in developing an immersion-compression test. They used the same source of Colorado aggregate as used in the test highway and compared results with a Potomac river sand and gravel of known quality. Initially they performed DIS tests on the aggregates under study. After testing, the Colorado aggregate showed only 40 to 50 per cent of its surface area had retained its asphalt coating, while 95 to 100 per cent of the surface area of the Potomac River gravel remained coated. Using similar mixtures, cylindrical specimens 3 in. in diameter and 3 in. in height were molded at room temperature under a molding pressure of 1000 psi. This molding pressure was maintained for a 1 minute period.

Specimens from each mix were tested in compression after molding and after immersion in water for periods of 1 to 7 days at a temperature of 77 F. The Colorado aggregate mixture showed a retained strength from 0 to 5 per cent, while the Potomac River sand and gravel mixture had a retained strength ranging from 87 to 103 per cent. This method of test was considered so promising that its use was then extended to evaluation of other types of aggregates, filler, and asphalts.

Further development of the I-C test was reported by Pauls and Goode (12, 45). The sensitivity of the test was to be increased until the differences in hot mixtures in the laboratory agreed with field observations. They evaluated I-C retained strengths of bituminous mixtures by using: 1) a vacuum process to accelerate the saturation of the compacted mixture, 2) higher water bath temperatures, and 3) different immersion times.

In the vacuum process, the specimen was kept immersed in a water bath under 27 inches of mercury vacuum until air bubbles ceased to come from the surface of the specimen. This required about 20 minutes, then the vacuum was reduced to atmospheric pressure, which forced water into the empty voids of the specimen. It was determined by using swell and strength tests that no initial detrimental effect resulted from vacuum saturation. However, when comparative specimens were vacuum saturated and soaked in the regular manner there was a difference noted in their retained strength. The vacuum process was not severe for hot mixtures of the coarse-grained type but the fine-grained mixtures were affected to a much greater degree. It was concluded that the degree to which vacuum saturation affected values of retained strength was related to the fineness of the aggregate used in the mixture. Since fine-grained mixtures normally have a high resistance to the infiltration of moisture, and usually show good service behavior it was concluded that the vacuum process should not be used in the I-C test procedure.

A definite relation between temperature of immersion water and time of test was observed. This study evaluated the effect of immersion temperatures of 77, 100, 120, and 140 F, along with periods of immersion of 5 hours, 1, 4, 14, and 35 days. The best correlation between field service behavior and I-C results was obtained using 4 days immersion at 120 F. However, results of immersion tests at 140 F for 1 day and 120 F for 4 days showed a very close agreement. Therefore, results obtained with 1 day immersion at 140 F were considered acceptable.

The ASTM standard test designation: D 1075, for effect of water on cohesion of compacted bituminous mixtures was adopted in 1954 (9). In this test the bituminous mixture is evaluated by preparing 6 specimens, 4 in. in diameter and 4 in. high. The specimens are molded using a double plunger device, the final pressure of 3000 psi being applied for 2 minutes. The specimens are cured 24 hours at 140 F in air after which their bulk specific gravity is determined. The 6 specimens are then sorted into two groups so that the average specific gravity of each group is about the same.

Dry specimens are then brought to 77 F and tested in axial compression at a uniform rate of vertical deformation of 0.05 in. per minute for each in. of specimen height. Wet specimens are placed in a 120 F water bath for 4 days (alternate method is 140 F water bath for 1 day) then cooled in a 77 F water bath at least 2 hours before determining their compressive strength. The index of retained strength is calculated by dividing the compressive strength of the wet specimens by the compressive strength of the dry specimens.

A similar test procedure has been published by the U.S. Army Corps of Engineers (46). Eight Marshall specimens are molded for each test.

The standard size of specimen is 4 in. in diameter and 2.5 in. in height. Specimens are divided into two groups so that the average specific gravity of each group is essentially the same. Dry specimens are immersed in a 140 F water bath for not less than 20 minutes and their stability determined using a Marshall Stability procedure. Wet specimens are immersed in a 140 F water bath for 24 hours prior to determining their Marshall stability. This method measures the index of retained strength by dividing the wet Marshall stability by the dry Marshall stability. Mixes showing an index of retained stability of less than 75 per cent are rejected.

Swanberg and Hinderman (47) reported a variation of the ASTM immersion-compression procedure in which they used specimens 4 in. in diameter and 2 in. in height. The standard testing procedures were followed except the strength of the specimens were determined using the Marshall Stability Testing Head. Mathews et al. (48) also used this procedure in their 1965 work.

Olsen (49) reported on a study in which the I-C test was performed using both the ASTM test procedure (9) and the Marshall stability test procedure (46). It was observed that a higher percentage retained strength was obtained with the Marshall I-C method than with the ASTM I-C method. It was noted by Eager (50) that in comparing indices of retained stability, the method of compacting and testing the specimens must also be considered. Specimens molded by kneading action such as the Hveem Kneading Foot Compactor show significantly higher stabilities, as well as higher indices of retained stability, than specimens compacted by the ASTM standard double plunger, direct compression method.

There has been some criticism directed at the I-C test method. Sanderson (13) stated "The immersion-compression test is long, involved, and requires special apparatus not usually found in a highway engineering laboratory. The results of the test are somewhat dubious, even when performed with the best laboratory equipment." Goldbeck (51) noted there was not good correlation between the traffic test in the circular track and the standard I-C test.

Andersland and Goetz (52) reported the bituminous mixture containing hydrophilic rhyolite had a higher I-C retained strength, at the end of one day immersion at 140 F, than either the gravel mixture or the limestone mixture (reference their data in Table III). Mr. Paul Thompson, in the discussion to this paper, noted that he had observed quite dense specimens would not be completely saturated during the entire 4 days immersion time at 120 F. He recommended that the time for immersion of the specimens should be calculated from the time the specimens were entirely saturated by water, such saturation having been effected either by a vacuum saturation technique or by a preliminary water immersion for complete saturation.

One phenomenon reported by some investigators using the I-C test method deserves elaboration. This is the fact that in some instances the bituminous mixture being evaluated indicate a retained strength greater than 100 per cent. The question was asked Mr. Goode (53) if there were instances of higher compressive strength being obtained after water immersion. He replied "Oh yes, we quite frequently get a retained strength greater than 100 per cent, particularly with a mixture containing limestone aggregate." He theorized that the small amount of moisture absorbed by the test specimen actually created tensile forces between the coated particles of limestone aggregate within the specimen resulting in a higher compressive strength than would be obtained with a dry specimen.

Laboratory Test Tracks. Circular tests tracks have been developed to evaluate the durability of bituminous mixtures under the action of traffic. In 1936, Goldbeck (54) reported the results of investigations on single and double surface treatments, mixed-in-place construction, premixed cold laid pavements and asphalt concrete. The mixtures were compared on the basis of depth of rutting. Test track results were also related to field observations of similar mixtures. Temperature and moisture conditions were varied during the evaluation of track specimens. Goldbeck was of the opinion that stripping was apt to take place more rapidly in warm weather than in cold weather because the asphalt is softer and would more readily be stripped in this condition.

In 1949, Goldbeck (51) reported a comparison of immersion-compression tests results with laboratory traffic tests. Circular track stabilities and durability values were obtained by measurement of rut depths. The durability test track pavement was immersed 7 days at room temperature (65 to 75 F), then 20,000 passes of a 1000 lb wheel load were made with the track immersed. Six different asphalt concrete mixtures were tested, with immersion-compression retained strengths ranging from 71 to 105 per cent. All of the mixes were judged very durable from the test track loading results.

Holmes (34) also gave an account of the use of a circular test track used to evaluate the adhesivity of asphalt in bituminous mixtures. After compaction and curing of the bituminous mixture, the track specimen was submerged in water at 90 F and the asphalt-aggregate mixture was broken down by running the test wheels over the pavement surface. The number of revolutions of the machine required to loosen 10 per cent of the total mixture was chosen as the breakdown point. It was noted that duplicate test track results agreed within ± 15 per cent. Test track results were correlated with static immersion stripping test results and the adhesivity of various bituminous mixtures determined. Different additives were added to the asphalt to increase adhesivity. Holmes found the maximum improvement obtained with additives corresponded to a relative track life of 3.88 times that for untreated asphalt. He further observed that incorporation of lime into the bituminous mixture did not appear to alter the true adhesivity properties of the asphalt.

This work of Holmes was continued and enlarged by Klinger and Roediger (55). They attempted to establish whether a correlation existed between the SIS test, circular test track and field performance. It was concluded that relative service life of the pavement was not predicted by the static immersion stripping test results or the circular track test results. Additives increased the stripping resistance of the mixtures tested by SIS test and circular test track methods but no effect was evident in the pavements exposed to traffic.

An immersion wheel-tracking test developed to evaluate the significance of the traffic stresses in stripping of binder from aggregate was reported by Mathews and Colwill (56). The bituminous sample was immersed in water and subjected to the action of a reciprocating wheel running over its surface. The length of time for which the sample withstood this treatment without collapse was taken as the index of performance. The failure time for the 12 aggregates tested ranged from 1 minute to greater than 48 hours. Mathews et al. (48) later published the results of research work in which bituminous mixtures, using 16 different aggregates, were evaluated by the static, dynamic, boil, immersion-compression (strength by the Marshall Testing Head) and immersion wheel-tracking tests. These test results were compared with the road performance of the aggregates. An index of stripping was used by Mathews (57) to determine road performance, coupled with a 6 year field observation to determine when an aggregate exhibited appreciable stripping. It was concluded that the best correlation with road performance of the aggregate in bituminous-macadam was given by the immersion wheel-tracking test.

Other Test Methods. Several other procedures have been employed to evaluate stripping resistance of a bituminous mixture. In 1934, Stanton and Hveem (33) reported a swell test made on a compacted specimen of oil mixed with aggregate which represented the grading used in actual construction. The compacted mixture (4 in. in diameter by 2 in. high) was submerged in its mold for 24 hours and the amount of vertical swell determined. The permissible amount of swell was a function of the surface area of the aggregate in the mixture. They considered the swell test to be the most reliable method of determining the probable effect of moisture on the road surface.

Skog and Zube (42) report development of water susceptibility test used as a research method for studying the resistance of a bituminous mixture to moisture. A sample of the design mix was compacted with a kneading compactor, cured 75 hours at 140 F, subjected to moisture vapor passing up into the specimen and then tested for its Hveem stability and cohesion. The specimen resembled a standard Hveem specimen except for a 0.5 in. hole through its center. A minimum Hveem stability value for the mixture was specified.

An abrasion test to evaluate probable field performance of a bituminous mixture was reported by Swanberg and Hinderman (47). The compacted mixture, 2 in. in diameter and 2 in. high, were cured 24 hours at 140 F for 4 days. Specimens were then cooled and their saturated surface dry weight determined, then cooled to 35 F in a water bath. The test used a Deval abrasion machine filled with water at 35 F in which the specimen were placed and then the machine rotated for 33 minutes (about 1000 revolutions). The specimens were then weighed in a surface-dry condition and the percentage weight loss determined. Test results were compared with field performance of similar bituminous mixtures. They concluded that the abrasion test gave satisfactory correlation with field performance and reasonable agreement with immersion-compression test results.

Another abrasion test developed to subject the surface of a compacted bituminous mixture to dynamic water action along with simulated tire action was reported by Skog and Zube (42). A 4 in. diameter specimen held in its mold, after pre-conditioning, was clamped in a special shaking unit. Water and 4 solid rubber balls were placed on the mold and subjected to shaking for a 15 minute period. Water temperature was maintained at 100 F during the test. The weight loss of the specimen and visual estimate of the amount of stripping was then determined. Test results were correlated with different sources and grades of asphalt and different aggregate sources. It was observed that the abrasion loss varied with different asphalt sources, when the same aggregate type and source was held constant.

A non-destructive method of test for evaluating stripping resistance in compacted bituminous mixtures was reported by Andersland and Goetz (52). A sonic test method was employed using beam specimen 12 in. long by 2.5 in. thick. The test specimens were molded, cured and their initial sonic modulus of elasticity determined at 40 F in a saturated dry condition. Specimens were then immersed in tap water at a temperature of 140 F. At intervals of 1, 3, 5, 7, and 9 days the sonic modulus of elasticity of each specimen was determined. The percentage retained sonic modulus of elasticity was then calculated. Comparable results from the SIS test at 140 F and I-C test along with the sonic test results are shown in Table III. They concluded that the sonic test might produce laboratory results that would correlate well with field performance results. However, no field evaluation of the mixtures studied in the laboratory were reported.

## TABLE III

## STRIPPING RESISTANCE OF BITUMINOUS MIXTURES INDIANA AH TYPE SURFACE COURSE (After Andersland and Goetz, 52)

| Days Immer. | Static Immersion[1] (% Coated--140 F) | I-C (% Ret. Strength) | Sonic Modulus (% Ret. Modulus) |
|---|---|---|---|
| | Lafayette Gravel | | |
| 0 | 100 | 100 | 100 |
| 1 | 73 | 90 | 96 |
| 3 | 60 | 88 | 94 |
| 5 | 58 | 84 | 94 |
| 7 | 55 | 82 | 93 |
| 9 | 55 | 77 | 92 |
| | Greencastle Limestone | | |
| 0 | 100 | 100 | 100 |
| 1 | 77 | 91 | 94 |
| 3 | 73 | 84 | 92 |
| 5 | 70 | 77 | 91 |
| 7 | 70 | 72 | 90 |
| 9 | 70 | 64 | 89 |
| | Massachusetts Rhyolite | | |
| 0 | 100 | 100 | 100 |
| 1 | 17 | 93 | 96 |
| 3 | 12 | 81 | 91 |
| 5 | 12 | 68 | 80 |
| 7 | 12 | 60 | 74 |
| 9 | 10 | 28 | 71 |

[1] Static immersion sample sized to pass 3/8 inch sieve and be retained on 1/4 inch sieve.

# CHAPTER III

# ASPHALT CEMENT AND AGGREGATE MATERIALS

The asphalt cement utilized in this research was chosen to represent one of the more common binders used in asphalt pavement construction in Oklahoma. Physical properties of the asphalt were determined from standard test methods and include: penetration, kinematic viscosity, ductility, specific gravity, and thin film oven. The material was classified as a 85-100 penetration paving grade asphalt cement.

The mineral aggregates, hereafter called aggregates or rocks, selected for the study were representative of typical materials used or proposed for use in construction of Oklahoma highways. Eleven different sources were sampled and the aggregates include 3 types of limestone, 3 types of sandstone, one chert, and 4 types of gravel.

The physical properties of the aggregate that were evaluated include: specific gravity and absorption, Los Angeles abrasion, soundness, and acid insoluble. Each aggregate was identified as to its geological formation, age, and mineral composition, along with its petrographic description.

## Asphalt Cement

The sample of asphalt cement (penetration grade 85-100) was obtained from the Allied Materials Corporation plant at Stroud, Oklahoma. The asphalt was produced by physical subdivision using the steam and vacuum process of refining. Physical properties of the asphalt cement are shown in Table IV.

TABLE IV

PHYSICAL PROPERTIES OF ASPHALT CEMENT

| Characteristics | ASTM[1] Method | Test Value |
|---|---|---|
| Penetration, 77 F, 100 g, 5 sec | D5 | 93 |
| Ductility, 77 F, cm | D113 | 150+ |
| Viscosity at 275 F, Kinematic, cST | D2170 | 400 |
| Thin Film Oven Test | D1754 | |
| Penetration After Test, 77 F, 100 g, 5 sec | D5 | 60 |
| Percent of Original | | 64 |
| Ductility After Test 77 F, cm | D113 | 150+ |
| Average Weight Loss | D1754 | |
| Percent of Original | | +0.018 |
| Specific Gravity, 77/77 F | D70 | 1.003 |
| Softening Point, F | D2398 | 118 |
| Flash Point, F | D92 | 580+ |

[1] 1972 Annual Book of ASTM Standards, Part 11.

## Aggregates

The aggregates sampled are from a wide range of sedimentary rock types. The oldest rock is an Ordivician limestone; the chert is Mississippian in age; two limestones and two sandstones are Pennsylvanian in age; one sandstone and the conglomerate are Permian and the youngest three aggregates are Quaternary gravels. The aggregates are identified as to location, geologic unit, geologic age (period) and general aggregate classification and are shown in Table V.

The Oklahoma Highway Department (OHD) has performed standard physical tests on similar aggregates prior to using them in highway construction work. Records of the OHD were examined and representative test values for the physical properties of the aggregates obtained. These values are shown in Table VI. Chemical analysis of some rock types sampled were also available from the OHD and are reported in Table VII.

The aggregates that were chosen for study represent major sources of material available for highway construction in Oklahoma. The aggregate samples were secured by visiting each source and procuring a sample from stockpiles at the site. Approximately 150 lb of material of each aggregate type were obtained, representing a size gradation from the 3/4 in. to the No. 10 sieve.

The petrographic description of the limestones and sandstones are based on the study of thin sections and acid etched slabs by Willard McCasland. General quarry or pit descriptions of interest are given. The aggregates are identified by the town adjacent to their location. For purposes of description, the aggregates are divided into four groups: limestone, sandstone, chert, and gravel.

## Limestone

A limestone is a bedded sedimentary deposit consisting chiefly of calcium carbonate. Limestone is generally of marine origin and the remains of sea-living organisms may be common.

# TABLE V

# AGGREGATE IDENTIFICATION AND SOURCE

| Sample | County | Location[1] | | | Geologic Unit Period | General Classification |
|---|---|---|---|---|---|---|
| | | Sec. | Twp. | Rg. | | |
| Cooperton | Kiowa | 32 | 6N | 15W | Kindblade limestone<br>Ordovician | Limestone |
| Hartshorne | Pittsburg | 17 | 4N | 17E | Wapanucka limestone<br>Pennsylvanian | Limestone<br>(partly Siliceous) |
| Stringtown | Atoka | 16 | 1S | 12E | Wapanucka limestone<br>Pennsylvanian | Siliceous<br>Limestone |
| Cyril | Caddo | 36 | 6N | 10W | Rush Springs<br>Permian | Calcareous<br>Sandstone |
| Keota | Haskell | 23 | 10N | 23E | Bluejacket<br>Pennsylvanian | Siliceous<br>Sandstone |
| Onapa | McIntosh | 31 | 11N | 17E | Bluejacket<br>Pennsylvanian | Siliceous<br>Sandstone |
| Asher | Pottawatomie | 4 | 6N | 4E | Wellington-Admire<br>Permian | Chert Gravel[2] |
| Broken Bow | McCurtain | 4 | 7S | 26E | Alluvial Deposit<br>Quaternary | Siliceous<br>Gravel |
| Gore | Sequoyah | 19 | 12N | 21E | Alluvial Deposit<br>Quaternary | Siliceous<br>Gravel |
| Hugo | Choctaw | 36 | 5S | 17E | Terrace Deposit<br>Quaternary | Chert Gravel |
| Miami | Ottawa | 31 | 29N | 23E | Boone<br>Mississippian | Chert |

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[1] Based on USPLS Indian Meridian.

[2] From a loosely consolidated conglomerate.

## TABLE VI

## AGGREGATE PHYSICAL PROPERTIES

| Sample | Bulk Specific[1] Gravity | Absorption | L. A.[2] Abrasion | Soundness[3] | | Insoluble[4] (%) (+ No. 200 sieve) |
|---|---|---|---|---|---|---|
| | | | | NaSO$_4$ | MgSO$_4$ | |
| Cooperton | 2.67 | 0.8 | 24 | 0.8 | 4.4 | 1.2 |
| Hartshorne | 2.66 | 1.0 | 24 | 2.4 | --- | 23.3 |
| Stringtown | 2.57 | 0.5 | 22 | 4.4 | 6.3 | 72.8 |
| Cyril | 2.64 | 0.9 | 37 | 4.1 | --- | 59.2 |
| Keota | 2.48 | 2.4 | 40 | --- | --- | 96.3 |
| Onapa | 2.47 | 4.1 | 35 | 8.9 | --- | 92.1 |
| Asher | 2.46 | 3.2 | 25 | 6.5 | --- | 99.8 |
| Broken Bow | 2.69 | 1.3 | 25 | --- | --- | 98.3 |
| Gore | 2.68 | 0.6 | 29 | --- | 2.7 | 97.9 |
| Hugo | 2.52 | 1.8 | 20 | --- | 2.8 | 99.0 |
| Miami | 2.56 | 1.2 | 23 | 2.9 | --- | 95.4 |

[1] Reference ASTM Designation: C 127.

[2] Reference ASTM Designation: C 131.

[3] Reference ASTM Designation: C 88.

[4] Reference Oklahoma Test Method OHD-L-25 (Appendix).

## TABLE VII

## AGGREGATE CHEMICAL ANALYSIS

| Sample | Per cent by Weight | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | $CaCO_3$ | $MgCO_3$ | $SiO_2$ | $Al_2O_3$ | Total Fe (as FeO) | $P_2O_5$ | $K_2O$ | $Na_2O$ | S |
| Cooperton | 90.98 | 2.36 | 4.48 | 0.69 | 0.21 | 0.01 | 0.07 | 0.07 | 0.11 |
| Hartshorne | 73.60 | 1.84 | 21.20 | 1.48 | 0.79 | 0.07 | 0.14 | 0.08 | 0.12 |
| Stringtown | 30.47 | 0.58 | 62.42 | 2.36 | 1.95 | 0.13 | 0.12 | 0.03 | 0.23 |
| Cyril | 22.10 | 15.17 | 59.00 | 2.62 | 0.68 | 0.08 | 0.16 | 0.08 | 0.11 |
| Keota | 0.97 | 0.76 | 90.57 | 6.16 | 1.21 | 0.05 | 0.20 | 0.06 | 0.02 |
| Onapa | 2.18 | 1.65 | 83.00 | 10.56 | 1.63 | 0.08 | 0.33 | 0.06 | 0.02 |

Oklahoma has abundant limestone sources in some sections of the state. Since limestone was considered to be highly resistant to stripping, one limestone (Cooperton) of high purity was chosen for use as a basis for comparing relative stripping tendencies with other aggregates. Two other limestones of decreasing calcium carbonate content, i.e., decreasing purity, were also studied.

Cooperton. This material came from the folded limestones and dolomites of the Arbuckle Group that form hogbacks in northeastern Kiowa County. These hogbacks are generally known as the Limestone Hills Area of the Wichita Mountain Uplift. The quarry working face is about 210 feet high, out of a massively bedded limestone ridge which stands about 300 feet above the surrounding terrain. The structural dip of the formation was about 20 degrees north.

The rock is a gray to mottled gray and buff colored, very fine-grained, fossiliferous, hard limestone. The Cooperton specimen contains of 32 per cent pellets and oolites, ranging in size from 0.05 to 1.0 mm, with a median size of 0.35 mm. Grains are 100 per cent rounded, medium to tightly packed, well sorted and are cemented with sparry calcite. Micrite composes 30 per cent and dolomite 3 per cent. There are a few veins which are filled with calcite. The rock fractures along the calcite vein. There is no evidence of shale within the quarry site. The rock is classified as biopelsparite using the Folk (58) system and packstone using the Dunham (59) system.

Hartshorne. This aggregate is a gray to dark gray fossiliferous to cherty limestone, obtained from the Ouachita Mountains of Southeastern Oklahoma. The quarry has a 25 to 60 foot high working face, and the beds dip north-northwest at about 10 degrees. The upper 10 feet of rock has iron stain and is massively bedded. The lower 10 feet is thin bedded, with shale seams or laminae, overlying a 25 foot black, limey shale.

Three distinct types of limestone were observed. The dense limestone contains 95 per cent very fine grained spar (microspar) in the matrix and is classified as microsparite (58) or mudstone (59). Another section contains 63 per cent fossils, which are highly silicified. The fossils are very closely packed to almost touching, well to poorly sorted, and range in size from 0.05 to 4.0 mm. This specimen is classified as a pelmatozoan calcarenite (biosparite) (58), or packstone (59). The third section contains 89 per cent microspar, with 11 per cent fossils. Glauconite and quartz grains are scattered through the matrix. This specimen was classified as a calcarenitic micrite mudstone (58) or mudstone (59).

Stringtown. This sample consists of about 60 per cent tannish-gray to chocolate brown, fine-grained limestone, 30 per cent dark grayish-brown chert, and 10 per cent sandy shale. These were present in alternating layers about 1 in. in thickness.

The quarry is located southeast of the Choctaw Fault and northwest of the Tri Valley Fault in the frontal belt of the Ouachita Mountains. Beds are very steeply dipping to vertical in attitude. The working face of the quarry is about 240 feet high and is worked in 60 feet lifts.

Numerous veins, filled with calcite, are present at 0.2 inch to 1.0 inch intervals. The thin section contains two distinct units compositionally: one composing 80 per cent of the slide is chert and the other is dolomite. The crystals of dolomite are 0.04 to 0.2 mm in size and are loosely packed. The chert is very dense and is composed of cryptocrystalline quartz (chalcedony). The rock tends to fracture along the calcite veins. Minor amounts of pyrite are present in the specimen. The rock is classified by McCasland (60) as siliceous limestone.

## Sandstone

Sandstone is a sedimentary rock composed of noncarbonate grains 0.06 to 2.0 mm in diameter, which are cemented together in some fashion. The cementing material may be quartz, opal, calcite, dolomite, clay, or oxides of iron, either reddish (hematite) or yellowish (limonite). The colors are variable, white to gray, buff to dark yellow, and red to reddish brown are common (61). These colors depend largely on the nature of the cement.

According to McBride (62) many different classification systems for sandstone have been proposed, but none has been devised which adequately treats all of the important sandstone attributes. The sedimentary structure, texture, and composition of the sandstone are the three main characteristics used in their study. Composition is generally the most important feature for evaluation as a highway material.

Cyril. This quarry in the Anadarko Basin area has a working face of about 100 feet into the massively bedded sandstone, which contains some cross-bedding. This rock is mostly gray, with some red and yellow. It is a calcareous to dolomitic sandstone.

The quartz sand grains were well sorted, subangular to subrounded, loosely packed and cemented together with recrystallized microspar. The grains range in size from 0.02 to 0.3 mm, with a median size of

0.15 mm. Some of the grains were partially coated with hydrocarbon material. No veins were evident in the section. The quartz grains comprised 42 per cent of the section. The specimen is classified as a quartzose sandstone (quartzarenite) (62), with calcareous cement.

Keota. The quarry face consists of about 20 feet of gray, massive to thin-bedded sandstone, overlain by a brown thin-bedded sandstone, about 6 feet thick. The sample consists of 80 per cent quartz grains and 1 per cent feldspar bound together by a chert cement.

Grains are well sorted, subangular, closely packed, and range in size from 0.05 to 0.4 mm, with a median size of 0.2 mm. Grains appear corroded and sutured together. About 30 per cent of the sample has a distinct yellowish color or stain. Some thin laminae of organic material were noted. The specimen is classified as a quartzose sandstone (62).

Onapa. The quarry has a 20 foot working face, with 7 feet of grayish-tan, thin to thick-bedded sandstone underlain by interbedded shale and sandstone. The overburden is buff sandstone and gray shale, about 3 feet thick.

The specimen consists of 68 per cent quartz grains, and 31 per cent siliceous matrix (chert and chalcedony). Carbonaceous material, present as striations, comprises one per cent of the specimen. Grains are subangular, well sorted, and range in size from 0.04 to 0.20 mm, with a median size of 0.15 mm. The quartz grains are tightly packed and appeared corroded or etched by the matrix. The specimen is classified as quartzose sandstone (62).

## Chert

Chert is composed of cryptocrystalline quartz of various varieties. It is characterized by its hardness, conchoidal fracture, or splintery fracture if porous, and a variety of colors. The more dense rocks are very tough and are usually gray to black, or white to brown and have a waxy luster. The porous varieties have a chalky-like surface and are generally lighter in color; white or yellow, brown and red stain are common (63).

Miami. The sample of whitish gray chert was obtained from a stockpile of crushed waste material from the Eagle-Picher Zinc Mine. The material was excavated from massive chert beds, with 50 to 70 feet of working face in the mine. The aggregate contains 92 per cent chert, 6 per cent limestone and 2 per cent dolomite, zinc, iron and other trace metals. The crushed rock edges tend to be very sharp (60).

## Gravel

Gravel is a loose or unconsolidated coarse granular material, larger than sand grains. When such material is transported by running water it is sorted according to the strength of the current. In some cases beds are formed which consist approximately of equal sized particles. The particles which compose gravel are rocks fragments and individual minerals.

The form and appearance of these pebbles depend on the conditions of erosion, transportation, and deposition. Those which have experienced considerable transportation are likely to have a very smooth surface with a characteristics faintly dimpled, slightly dented appearance caused by their repeated collisions during movement. If the pebble is composite in nature, it commonly is pitted by weathering and removal of softer or more easily altered minerals (61).

Asher. This material was obtained from a pit which has a working face of about 15 feet in the Maud conglomerate. Overburden is 10 to 15 feet of sandstone. Large aggregate (1.5 in. and above) is separated at the crusher for use as decorative stone.

The aggregate is a whitish pink to brown, fairly homogeneous gravel, with some hematite stain. An analysis indicates that 94 per cent of the sample is banded chert and 6 per cent is cherty limestone, with some hydrocarbons present as a surface coating. The material is classified as a chert gravel (60).

Gore. This material is from a deposit in the Arkansas River, where it was obtained by dragline operations. The excavating procedure secured material which is fine sand to cobble size.

The aggregate pieces are multicolored, heterogeneous and subangular to rounded before crushing. Mineral and rock analysis of the sample indicates that it is 59 per cent quartz, 22 per cent chert, 10 per cent granite, 8 per cent feldspar, and 1 per cent sandstone. The material is classified as a siliceous gravel (60).

Broken Bow. This sample was obtained from the flood plain adjacent to the Mountain Fork River. The pit was 25 to 30 feet deep and was worked by dragline. Soil overburden averaged 5 feet in depth. Boulders, cobbles, and sand represent the sizes of material obtained from the pit.

The gravel is 50 per cent quartz, 24 per cent chert, 21 per cent quartzitic sandstone, and 4 per cent metamorphic rocks. The aggregates are subrounded prior to crushing operations. The material is classified as a siliceous gravel (60).

<u>Hugo</u>. This material is a terrace deposit of the Kiamichi River. The pit excavation was operating on a 10 feet deposit of sand and gravel, which had been covered with 2 to 4 feet of soil overburden. The aggregate is 93 to 95 per cent chert, 5 to 7 per cent sandstone, with traces of quartz, ironstone, and feldspar. The aggregate is a homogeneous mixture of brown chert rocks, which are well rounded before crushing. The aggregate is classified as a chert gravel (60).

# CHAPTER IV

# LABORATORY TEST PROCEDURES AND EQUIPMENT

The immersion-compression test procedure adopted was patterned after the ASTM standard method (9). However, several variations from this standard method were used to take advantage of available molding and testing equipment. The specimens were molded with a motorized gyratory-shear compactor, rather than using the static double plunger compression method of ASTM. Instead of the ASTM method of testing in block compression, the specimens were tested in axial compression, across the diameter, using a Marshall Stability Testing Head. The specified ASTM specimen height to thickness ratio is about 1.0, while the specimens tested in the Marshall head had a height to thickness ratio of about 2.0.

The static immersion stripping (SIS) test procedure conformed to the ASTM standard procedure, using a water immersion temperature of 77 F. A modified static immersion stripping test was also used where the immersion water temperature was increased to 140 F. The dynamic immersion stripping (DIS) test used the same sample preparation and coating technique as employed in the static immersion stripping test. The DIS test procedure and evaluation method was designed to obtain a relative stripping factor for each aggregate tested.

The surface reaction test was devised to give a quantitative measure of stripping. The stripped specimen to be evaluated was obtained from the DIS test. A duplicate, uncoated, specimen was used to estimate the original total surface area of the aggregate.

## Immersion-Compression Test

### Mix Design Method

The mixture for the immersion-compression (I-C) specimens was designed to conform to an OHD Type B bituminous mix (64). This is a fairly coarse graded mix used for both surface and base courses. Gradation of the mixture was controlled by sieving the aggregates into eight different sizes on U.S. Standard Sieves and then recombining to obtain the required gradation.

The coarse aggregate used in the mixtures, size 3/4 in. to the No. 10 sieve, was obtained as previously described in Chapter III. The fine aggregate (passing No. 10 sieve) was obtained from the Arkhola Sand and Gravel plant at Muskogee, Oklahoma. The material had been dredged from the Arkansas River and was a high quality quartz sand. Dust or mineral filler (passing No. 200 sieve) used as part of the fine aggregate was obtained from the Cooperton limestone screenings. The eleven different coarse aggregates were combined, in sequence, with the fine aggregate. Each mixture was identified by the name of the coarse aggregate used. The combined grading of the aggregate mixture along with the OHD Type specification limits are shown in Figure 3. Using the mid-point gradation as indicated, yielded a very dense compacted mixture.

Optimum asphalt content for each aggregate mixture has previously been determined using the standard OHD method of design. The optimum asphalt contents ranged from 4 to 5 per cent. For the purpose of

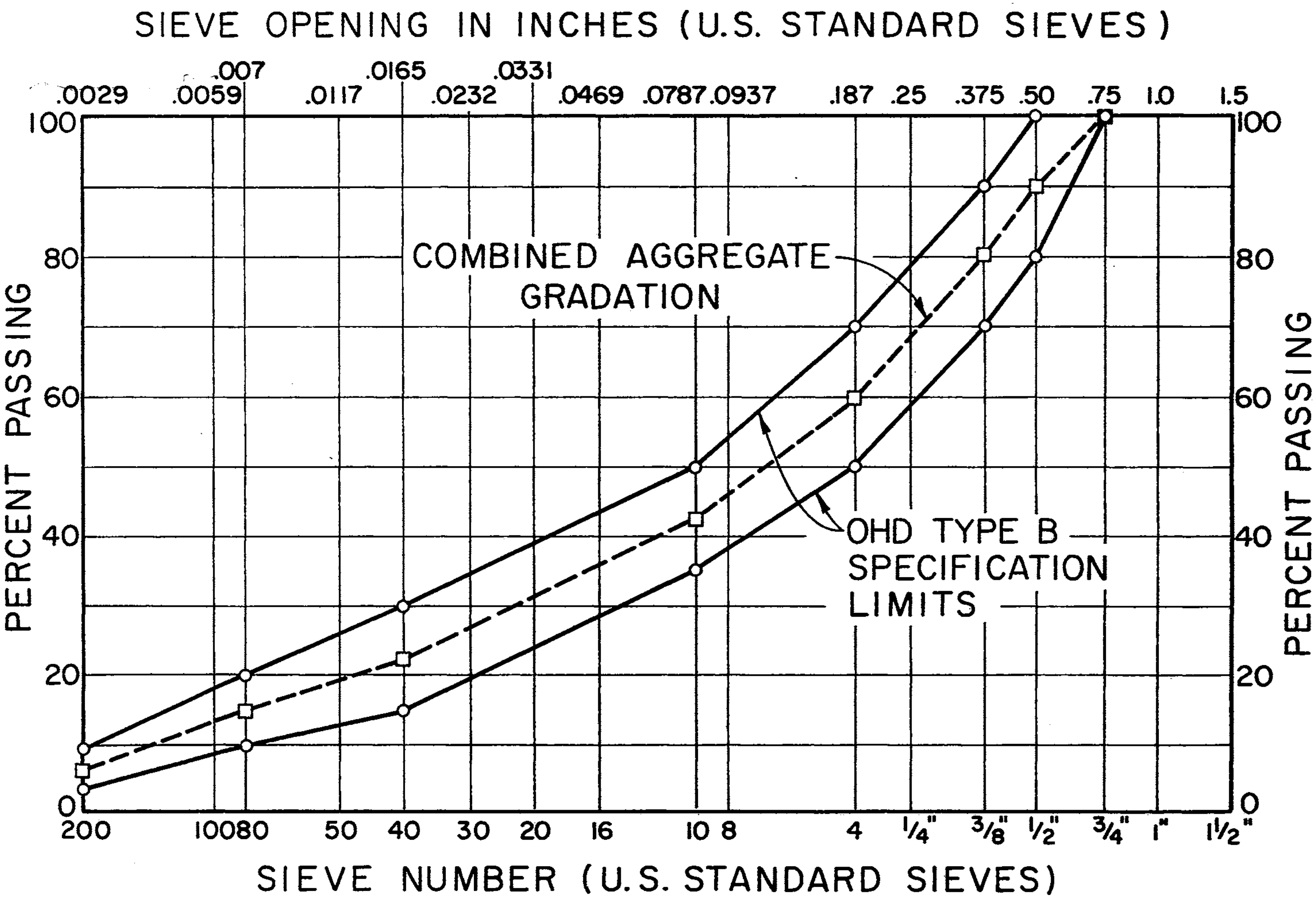


Figure 3. Combined Aggregate Grading Chart

obtaining comparable immersion-compression data, 4 specimens were molded at 4 per cent asphalt content and 4 specimens were molded at 5 per cent asphalt content. This procedure was repeated for each of the eleven coarse aggregates.

## Mixing and Molding

The blended aggregate mix and asphalt cement were heated to a temperature of 325 F. The aggregate was placed in the mixing bowl and the proper amount of asphalt cement added. The mixing was accomplished for each individual specimen using a Hobart Mixer. About three minutes of mixing time was required to fully coat the aggregate with asphalt. The mixture was then placed in a holding oven to obtain a molding temperature of 260 ± 5 F.

The specimens were molded using the motorized gyratory-shear compactor as shown in Figure 4. This method has been standardized by the Texas Highway Department (Test Method Tex-206-F, Part II). A total of 168 specimens were molded and tested during the preliminary investigation before a molding procedure for the I-C specimens was developed.

The standard molding procedure was modified for the immersion-compression specimens by gyrating the mixture one cycle (3 revolutions) under a load of 50 psig, rather than gyrating the specimen at 50 psig pressure until the end point of 100 psig pressure was obtained with one full stroke of the hydraulic jack. A leveling pressure of 800 psig was then used to obtain final compaction. The standard procedure requires a leveling pressure of 2500 psig.

This modified molding procedure was then used in preparing all of the immersion compression test specimens. After compaction, the

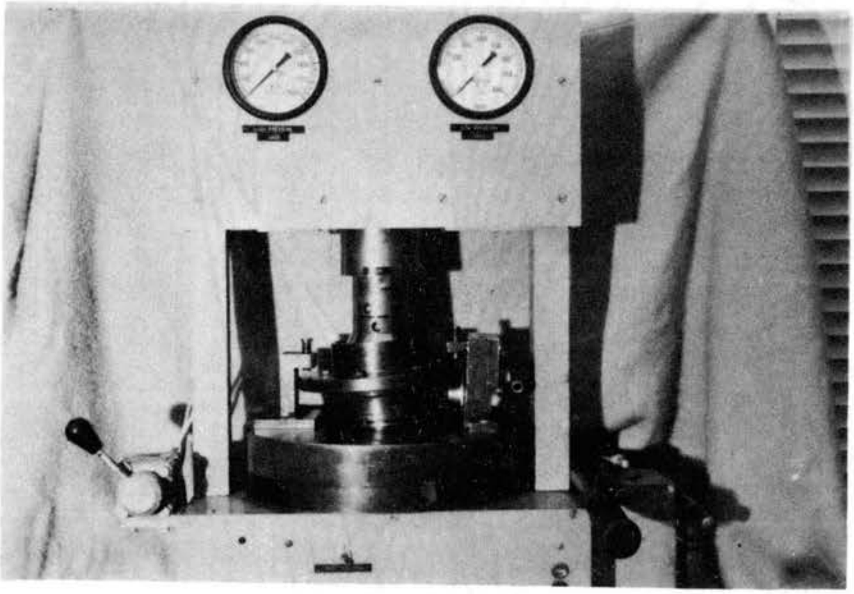


Figure 4. Motorized Gyratory-Shear Compactor

specimens had a 4 in. diameter and were approximately 2.1 in. high. The specimens were removed from the mold by hand pressure. After cooling to room temperature the specimen height was determined using the average of 5 measurements, with the device shown in Figure 5.

Bulk specific gravity of the I-C specimens was obtained by weighing in air, then weighing in water. The specimen was allowed to remain in water only a short period of time while obtaining this weight.

Each group of specimens was divided into wet and dry sub-groups such that the bulk specific gravity of the specimens in each sub-group were approximately equal. After grouping, the specimens were then cured 24 hours in a 140 F oven.

### Curing and Vacuum Saturation

Preliminary investigations revealed that the mid-point gradation of the OHD Type B mixture was a relatively dense mixture and that the specimens were not fully saturated when soaked in a 140 F water bath for 24 hours. Previously published research by Pauls and Goode (12) showed that vacuum saturation of open graded mixtures resulted in no detrimental effects to the specimen. Therefore, it was deemed desirable to vacuum saturate the wet specimens to insure there was water available to provide an opportunity for stripping.

The vacuum saturation process used was based upon work done by Manke (65). The wet specimens were placed in a vacuum dessicator, and the air evacuated for 10 minutes, the specimens were flooded with deaired distilled water and the vacuum process contained an additional 10 minutes, using a vacuum of 29 inches of mercury. Application of the

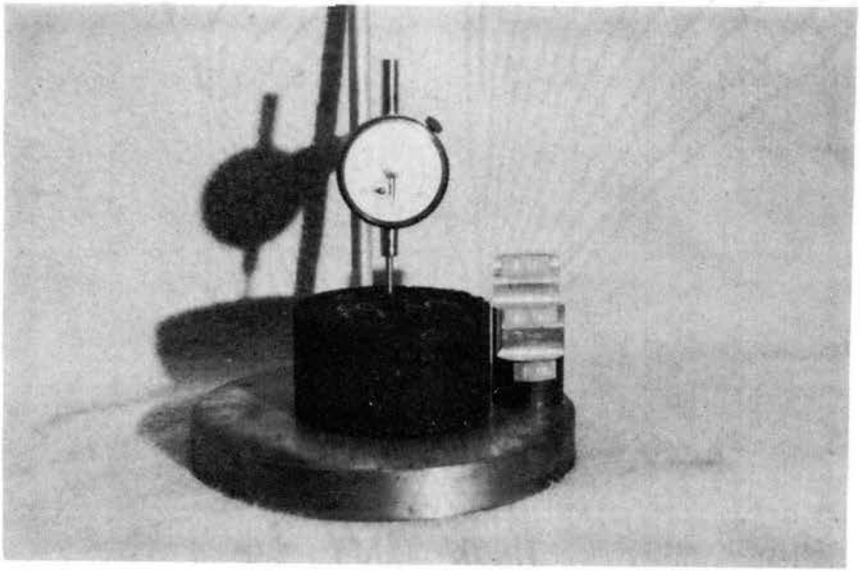


Figure 5. Compacted Specimen Height Measuring Device

vacuum was then discontinued and the system opened to atmospheric pressure forcing water into the void spaces.

The saturated surface dry weight of the wet specimens was then obtained to determine the amount of water absorbed. These specimens were then immersed for 24 hours in a 140 F distilled water bath.

The dry specimens were placed in plastic, water tight, bags and cured at room temperature for 24 hours. The wet specimens and dry specimens (in their water proof bags) were placed in a 77 F water bath 2 hours prior to their compressive test.

### Immersion-Compression Testing

The ultimate strength of each specimen was determined using a Marshall Stability testing head. The arrangement of the specimen and Marshall testing head in the testing machine is shown in the photograph of Figure 6. Loading rate of the testing machine was adjusted for 0.2 inch per minute. The compressive strength of each specimen was determined by dividing the maximum load obtained by the specimen cross-sectional area. The percent of retained strength of each group of specimens was determined by dividing the average strength of the wet specimens by the average strength of the dry specimens and multiplying by 100.

## Static and Dynamic Stripping Tests

### Sample Preparation

Each of the eleven coarse aggregates being evaluated in this work had been previously sieved into different sizes for the I-C mixtures.

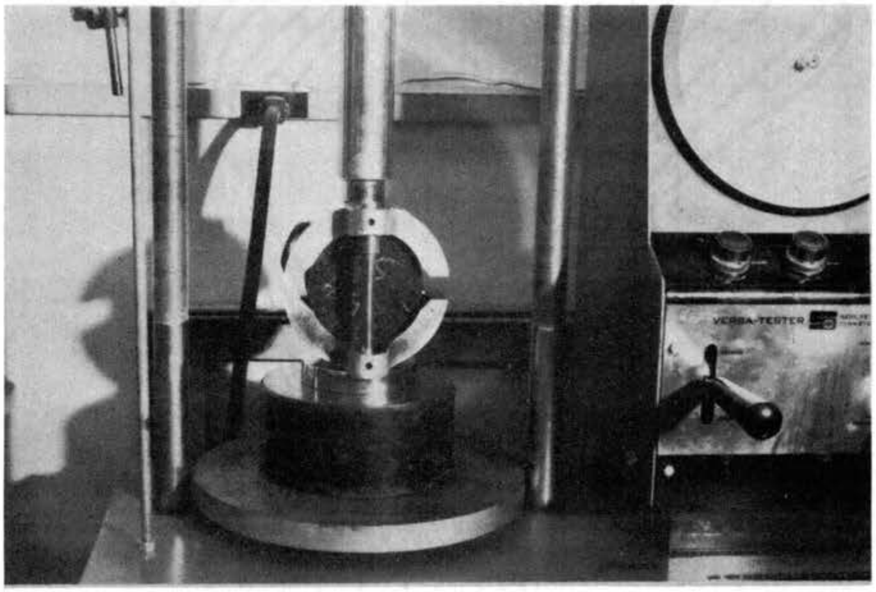


Figure 6. Immersion-Compression Test Using the Marshall Stability Testing Head

The 3/8 inch to No. 4 size material was quartered and resieved to obtain approximately 2000 g of material passing the 3/8 inch sieve and retained on the 1/4 inch sieve.

The aggregate sample was then washed, oven dried, and quartered to obtain representative samples of approximately 100 g each. Exactly 100.0 ± 0.2 g of the dry aggregate was weighed and placed into large aluminum moisture boxes for storage until required in the testing work. The number of individual rock particles in each 100 g lot was counted. Ten samples of each aggregate under study was prepared in this manner. The 10 samples were used, in a random manner, in performing these tests: 2 specific gravity and absorption tests, 2 static immersion stripping tests, 2 dynamic immersion stripping tests, and 2 surface reaction stripping tests. The remaining 2 samples were held in reserve.

## Coating

The aggregate and asphalt cement were heated to 250 F prior to the coating operation. To each of the 100 g samples of aggregate, 6 g of asphalt was added. The mixture was stirred and manipulated with a spatula until each rock was coated with asphalt. A hot plate was used to heat the mixture, as required to achieve 100 per cent coating. About 3 minutes of hand mixing time was ordinarily required. The gravel aggregate samples (Asher, Broken Bow, Gore and Hugo) were much easier to coat with asphalt than the crushed aggregate samples.

The individual particles of asphalt coated rock were placed in a pan of cold distilled water after mixing. Cold water was necessary to prevent the coated rocks from sticking together.

## Static Immersion Stripping Test

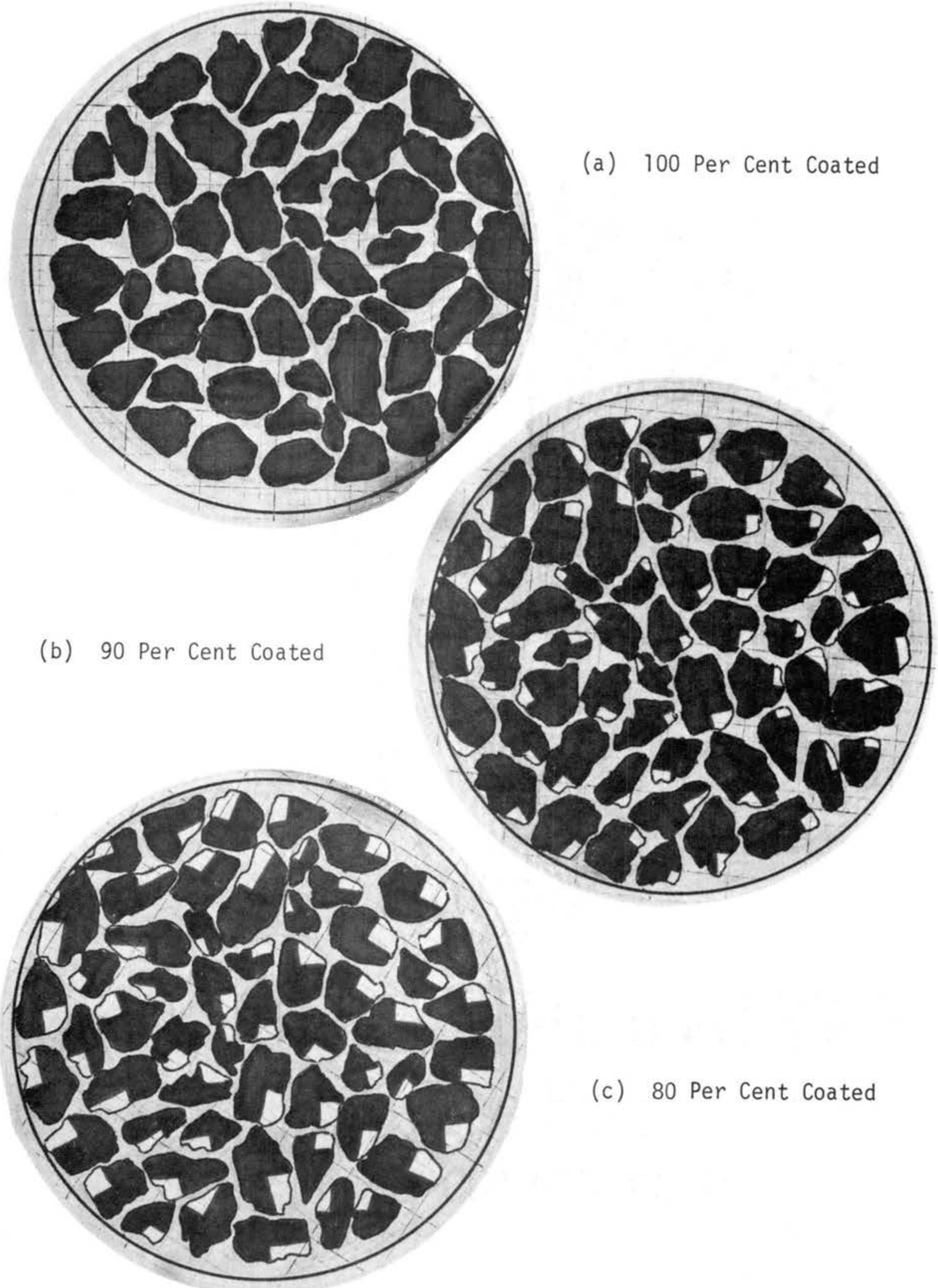
The sample preparation and coating procedure as given above follows the standard method of test for coating and stripping of bitumen-aggregate mixtures, ASTM Designation: D 1664 (8). After cooling in the chilled water the coated sample was placed in a glass jar and covered with 600 ml of distilled water. The jar was capped and placed, partially submerged, in a 77 F water bath, and left undisturbed for 18 hours.

The amount of stripping was then visually estimated, using the ASTM standard procedure. To facilitate this evaluation, a comparison graph or chart was prepared. This chart was prepared by tracing the outline of typical aggregate particles inside a circle the same diameter as the glass jar in which the samples were immersed. The cross sectional areas of the aggregate particles were darkened to represent different amounts of coated surface. This comparison chart is shown in Figure 7.

No stripping of any of the various aggregates was observed when coated with asphalt cement and subjected to the static immersion stripping test at 77 F. With a longer period of immersion or higher immersion temperatures, it was anticipated that some stripping of the aggregates would occur. Therefore, the SIS (77 F) samples were then placed in a 140 F water bath and left undisturbed for 18 hours. The amount of stripping (which was considerable) was then visually estimated, using the comparison chart of Figure 7. This test method was designated the static immersion stripping test at 140 F.

## Dynamic Immersion Stripping Test

In order to accelerate the stripping action of water on coated aggregate a dynamic stripping device was constructed. The literature

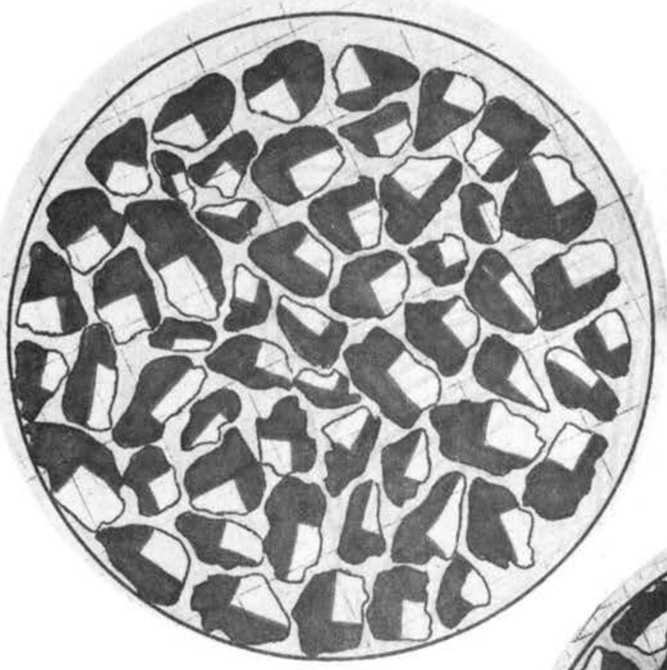


(a) 100 Per Cent Coated

(b) 90 Per Cent Coated

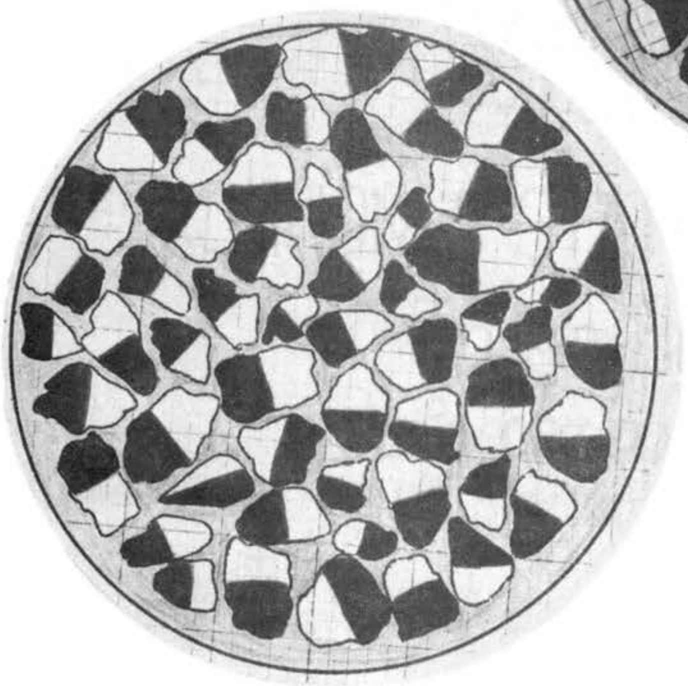
(c) 80 Per Cent Coated

Figure 7. Visual Estimation Reference Chart



(d) 70 Per Cent Coated

(e) 60 Per Cent Coated



(f) 50 Per Cent Coated

review showed many previous investigators had used a dynamic immersion stripping (DIS) test to evaluate the effects of water on asphalt coated aggregate (13, 15, 17, 20, 23, 29, 30, 32, 48). The method originally used by Nicholson (17) was followed in this study.

An apparatus was designed and built to hold six glass jars of approximately one quart capacity. The device was rotated about a horizontal axis at about 40 rpm. This caused the coated aggregate sample to fall from one end of the jar through the water to the other end during each revolution. A photograph of this dynamic stripping device is shown in Figure 8.

Preliminary tests using the DIS device revealed that the non-stripping aggregate (Cooperton limestone) would partially strip when the sample was tumbled continuously for 4 hours. The hydrophilic (siliceous) aggregate particles, also retained more than 50 per cent of their coating at the end of 4 hours of tumbling. Therefore, a 4 hour DIS test period was chosen, with the temperature maintained at about 68 F, which was the normal laboratory temperature.

The dynamic immersion stripping test procedure involved coating the aggregate with asphalt cement as was done for the static immersion test, then subjecting the coated aggregate particles to 4 hours of water agitation. The visual estimate of the amount of stripping was made at the end of 1 hour, 2 hours, and 4 hours. This evaluation method was similar to that used in the static immersion test, using the comparison chart of Figure 7.

It is noted that the intended use of the dynamic immersion stripping test was to subject each aggregate sample to the same effect of agitation in water, and then to compare the visually estimated relative

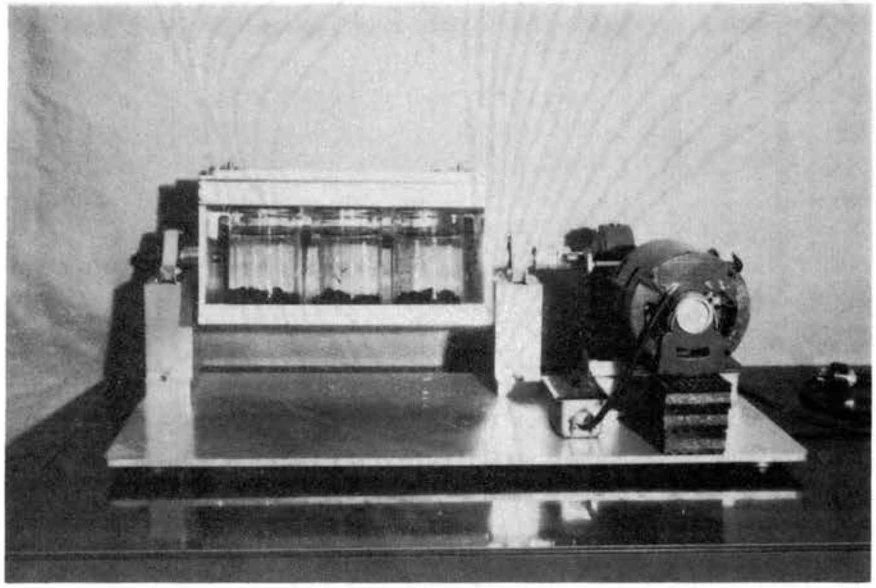


Figure 8. Dynamic Immersion Stripping Machine

stripping tendency of each different type of aggregate. The same stripped aggregate sample was used in the Surface Reaction Test, where a more quantitative evaluation of the amount of stripping that occurred was obtained.

## Surface Reaction Test

The objective of this test was to evaluate in a more quantitative manner the amount of surface stripping undergone by an asphalt coated aggregate sample. The technique employed was based on the following hypotheses:

1) The exposed surface area of the aggregate is proportional to the change in gas pressure resulting from the reaction between a suitable reagent and the aggregate surface.

2) An asphalt cement coated aggregate will not react with the reagent and create a significant pressure.

Development of the surface reaction test (SRT) required: a suitable reagent, equipment to measure the reaction gas pressure and temperature, a suitable test procedure, and evaluation of the relationship between aggregate surface area and gas pressure generated.

### Reagents

A suitable reagent for this work is defined as one that, when placed in intimate contact with an aggregate surface, will cause a chemical reaction creating a measurable gas pressure. A reagent strength was desired such that the resulting chemical reaction would not be so violent as to deeply etch the aggregate surface.

There was concern, that with sufficient time to react, a strong reagent would not only dissolve the exposed surface of the aggregate, but would continue to react and undermine adjacent asphalt coated areas of the aggregate surfaces. The resultant increase in gas pressure would cause an erroneous determination of the exposed aggregate surface area. This problem was minimized by using the weakest reagent solution that would create a measurable change in gas pressure and measuring this change in gas pressure in the minimum possible time.

Limestone Aggregate. Preliminary laboratory work indicated that the calcium carbonate ($CaCO_3$) in the limestone would react in the desired manner using about 1.0 normal hydrochloric acid (HCl). A 100 g sample of aggregate, when reacted with 200 ml of 1.0 normal HCl acid solution would create between 4 and 10 psi of gas pressure. The acid solution would be depleted in about 10 minutes of reaction time at room temperature. Carbon dioxide ($CO_2$) is the gas generated in this reaction.

Siliceous Aggregate. For aggregate composed mainly of silicon dioxide ($SiO_2$) the reagent required to obtain a measurable gas pressure was concentrated hydrofluoric acid (HF). The reaction creates noxious silicatetrafluoride gas ($SiF_4$). This acid and gas are highly toxic to humans and must be handled very carefully. All work with hydrofluoric acid was carried out in a well ventilated fume hood, using appropriate safety equipment. Preliminary work indicated that although the $SiF_4$ pressure was small, it was of sufficient magnitude to be measured.

Mixed Composition Aggregate. These are aggregate containing appreciable amounts of both $CaCO_3$ and $SiO_2$, and other constituents. The reagent desired was one that would react with both types of chemical compounds. Preliminary work indicated that a mixture of HF and HCl would create a measurable gas pressure when reacted with aggregate of mixed composition. A 200 ml acid solution was composed of: 27 ml concentrated hydrofluoric acid, 54 ml concentrated hydrochloric acid, and 119 ml of distilled water.

### Equipment

This method of test required the measurement of the gas pressure generated when a aggregate sample is inundated with a suitable acid. Since the temperature of the reaction affects the volume of the gas, it was necessary to measure and record simultaneously the pressure and temperature involved in the reaction.

The device developed to accomplish this gas pressure and temperature measurement consisted of modifying a six quart pressure cooker, and equipping it with suitable instrumentation to measure and record simultaneously the pressure and temperature.

Recorder. A two-arm recorder was used for recording both temperature and pressure at the same time. A Sargent Model DSRG Recorder, accurate to 0.25 per cent or 5 microvolts and with a reproducibility of 0.1 per cent, was utilized. The recorder had three chart travel speeds available, 0.5, 1.0 and 2.0 inches per minute.

The Sargent Recorder used was an automatic, self-balancing potentiometer, which graphically records measured potential as a function of time. This potentiometer is used conventionally for most accurate measurements since it utilizes a null balance principle which permits measurement without withdrawing power from the system being measured.

Pressure. The pressure was measured using a Stratham Model PA 300TC-30-35 pressure transducer with a range of 0 to 30 psig. This transducer is a strain-gage pressure device which converts a pressure into change in resistance due to strain. A diaphragm of magnetically permeable stainless steel is clamped between two blocks, and deflects when a pressure difference is applied. The deflection is proportional to the pressure differential across the diaphragm.

The pressure transducer output voltage to the recorder varied with the pressure differential across the diaphragm. The pressure transducer-recorder relationship was obtained by a calibration process.

The pressure transducer was calibrated from 0 to 5 psig using a Master Test Gage, type 210-C, manufactured by the Marsh Instrument Company. The recorder was set to zero with zero pressure on the pressure transducer. Then 5 psig was applied to the pressure transducer, using the test gage, and the recorder pen was made to travel full scale across the graph paper. The graph paper used on the recorder was divided laterally into 200 divisions, therefore each division on the chart was equal to 0.025 psig.

Temperature. The temperature of the gas generated in the pressure vessel was monitored by use of a thermistor. A thermistor is a semiconductor which exhibits large change in resistance with slight change in temperature. The thermistor used in this work was a YSI Model 44004 manufactured by the Yellow Springs Instrument Company.

The thermistor was linked to a YSI Model 47 Scanning Tele-Thermometer, which converted the change in resistance of the thermistor into temperature. In turn, the Tele-Thermometer was connected to the Recorder such that continuous temperature readings were obtained. The recording pen was adjusted, such that temperature scale ranged from 15 C to 100 C, and one division on the graph or chart paper equaled 0.5 C.

Pressure Container Device. As stated earlier, a six quart stainless steel pressure cooker was modified and used as the pressure vessel. The complete apparatus is shown in the photograph of Figure 9.

The pressure transducer was centrally mounted on the removable top of the pressure vessel, the temperature probe cavity extended through the top about one inch and was just large enough in diameter to contain the thermistor. A stainless steel pressure release valve was also mounted on the top. This valve was used in calibration of the pressure transducer and to release the pressure in the vessel at the completion of the test.

Since the pressure vessel was to be operated as a closed system, it was necessary to provide a means of adding the acid to the aggregate without changing the ambient pressure. A 250 ml stainless steel beaker was attached to a rod extending through the body of the pressure vessel. The rod was sealed off by using neoprene o-rings both inside and outside the wall of the pressure vessel.

A handle was attached to the exterior end of the shaft. To inundate the aggregate specimen the beaker was positioned upright and filled with 200 ml of the acid solution; then the handle was turned until the contents of the beaker were poured into the plastic liner that held the aggregate sample. The beaker was made removable from the end of the shaft by use of a threaded joint. Details of the pressure container apparatus are shown in Figure 10.

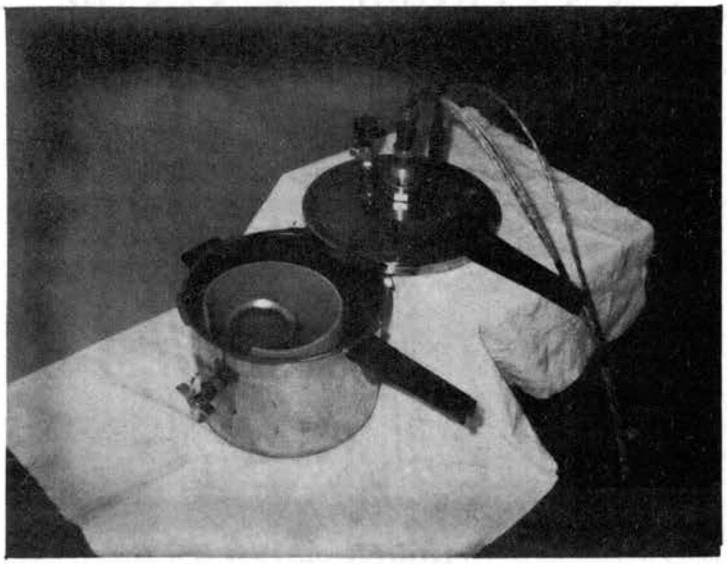


Figure 9. Pressure Container Device

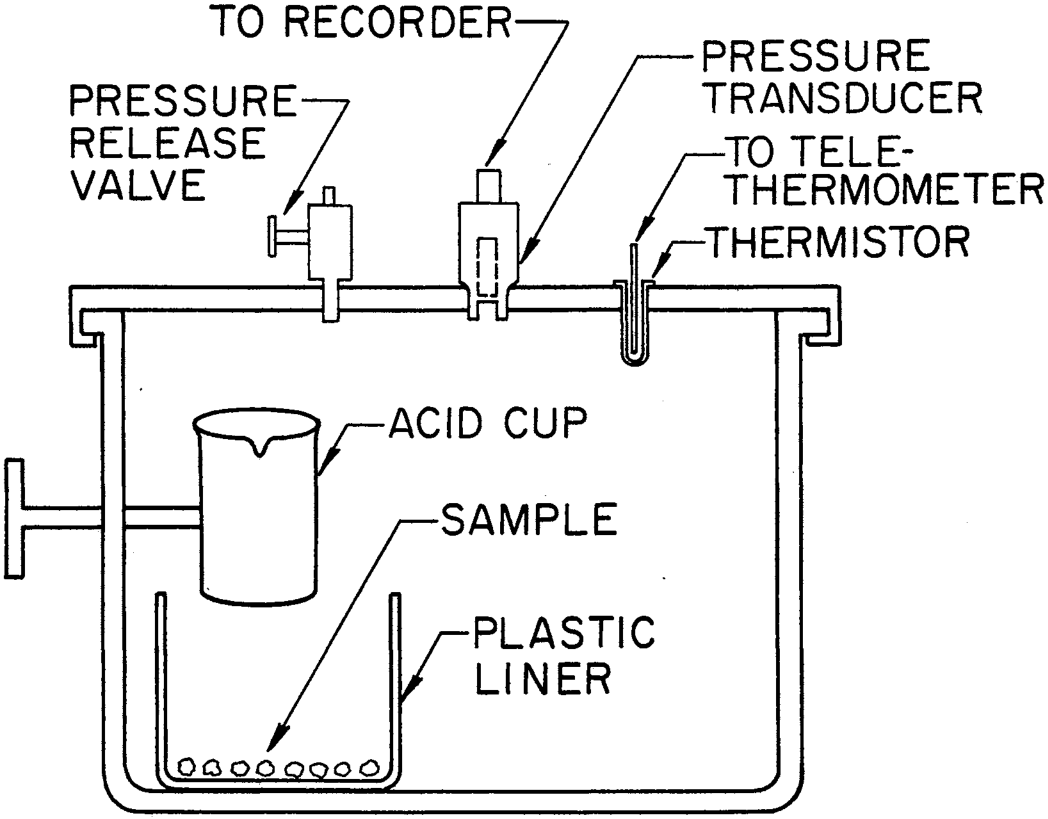


Figure 10. Details of Pressure Container Device

Materials to be used in the pressure apparatus were somewhat of a problem. The reagents proposed for use to obtain the desired surface reaction with the aggregates were hydrochloric acid and hydrofluoric acid. Hydrofluoric acid is a powerful, corrosive poison. It boils at 19.4 C and the vapor is quite as effective as that of the acid. Its aqueous solution dissolves glass, reacting with the silica to form silicon tetrafluoride. HF is used in various concentrations for frosting, etching and polishing glass, and for removing sand from metal castings. Because of its solvent properties it was used in this research. Hydrochloric acid will dissolve such metals as sodium, iron, tin and zinc. Stainless steel is one of the more resistant metals to the effects of hydrochloric acid and hydrofluoric acid. It was used for all the component parts of the pressure apparatus which were exposed to the acid solutions and gases generated during the surface reaction tests.

To reduce contact between the acid solution and the pressure vessel, a small plastic container was used to hold the aggregate-acid mixture during the reaction period. Plastic materials which are resistant to the effects of HCl and HF include both polystyrene and polyethylene. The plastic container was of polyethylene material. The size of the plastic container was selected such that a 100 g aggregate sample would be completely inundated by the 200 ml acid solution.

## Test Procedure

Sample Preparation. Half of the aggregate samples to be evaluated had previously been coated with asphalt cement and partially stripped in the dynamic immersion stripping test. An uncoated duplicate sample of the aggregate was immersed in distilled water at the same time as the DIS specimen was immersed. At the end of the DIS test, the partially stripped and uncoated samples were dried by blotting with paper towels and spread out in pans to air dry. The samples were air dried approximately 24 hours before initiating the surface reaction test.

It was desirable to perform this test on oven dry materials, however, when the partially stripped aggregate samples were oven dried at 212 F, the remaining asphalt cement diffused and completely recoated the stripped aggregate surfaces. Because of this recoating tendency, oven drying was eliminated and the samples were simply air dried before testing.

Calibration of Equipment. Prior to beginning a series of tests, the strip chart recorder was checked to ascertain proper operation. The pressure was checked with the Master Test Gage, while the thermistor and Tele-Thermometer temperatures were checked with a precise thermometer.

Reagent. Each test required 200 ml of acid solution. Duplicate samples were tested, i.e., 2 uncoated and 2 partially coated samples of each of the various aggregates. A liter of acid solution was prepared for use in testing each different aggregate sample.

The acid test solutions were prepared using reagent grade acids and deaired distilled water. All proportions were calculated on a weight basis. Quantities of HCl were measured using a graduated cylinder. Water and HF were added to solutions by weight. The liter of acid solution was mixed by tilting the bottle back and forth several times and then allowing it to cool overnight.

The 200 ml of acid solution to be used in each test was measured by a graduated cylinder and placed in a 250 ml nalgene jar and tightly capped. The weight of acid solution in each jar was determined and its density, in g/ml, was calculated. This density and weight determination was used as a check for obtaining equal amounts of solution for each test. The balance of the liter of solution originally prepared was retained for titrations.

The actual normality of the acid was determined by titration against a known weight of sodium carbonate using methyl orange as an indicator (66). All work with the acid solutions containing HF was carried out using polyethylene or polystyrene containers.

Performance. The pressure container was connected to the recorder and Tele-Thermometer by about 15 feet of electrical wiring. Hood temperature was maintained about 68 F. Before initiating a test, samples to be tested, acid solutions and the pressure container were all placed in the fume hood and brought to a constant temperature. The recorder and Tele-Thermometer were placed adjacent to the fume hood. The test procedure was as follows:

1) The pressure valve was opened and the top of the pressure vessel removed.

2) The sample to be tested was placed into the pressure vessel, in its plastic container, and was positioned beneath the acid beaker.

3) The beaker was installed in the pressure vessel, leveled, and the 200 ml of acid solution poured into the beaker.

4) The lid was placed on the pressure vessel, the pressure recording pen was set to zero, the chart speed was set at 1.0 inch per minute and the power switch turned to "Record" to start the chart drive.

5) The pressure release valve was closed on the pressure vessel, and a stopwatch started simultaneously with the instant the acid was poured onto the aggregate.

6) The reaction was monitored by observing the pressure and temperature traces on the recorder.

7) At the completion of the test, which ordinarily lasted 5 minutes, the pressure release valve was opened and the recorder chart drive turned to "Standby."

8) After the pressure was released, the top of the pressure vessel was removed and the acid beaker taken out. The sample was removed and the reaction of the acid solution and sample was terminated.

a) For samples tested with HCl this was accomplished by flooding the mixture with tap water.

b) For samples tested with HF the reaction was stopped by slowly adding a sufficient amount of calcium oxide slurry to deplete the HF in the mixture. Methyl orange indicator was used to determine when the solution was neutralized.

9) The sample was then washed over a No. 10 sieve, air dried 24 hours, and its final weight determined.

10) The acid beaker and plastic sample container were then washed and dried prior to initiating another test.

The Tele-Thermometer, recorder and pressure vessel are shown in Figure 11a. The pressure vessel had been removed from the fume hood for the photograph. The photograph of Figure 11b contains a typical pressure-time curve for an uncoated limestone aggregate sample.

Stripping Evaluated. The pressure-temperature curves plotted on the recorder chart were then analyzed. A horizontal line was drawn

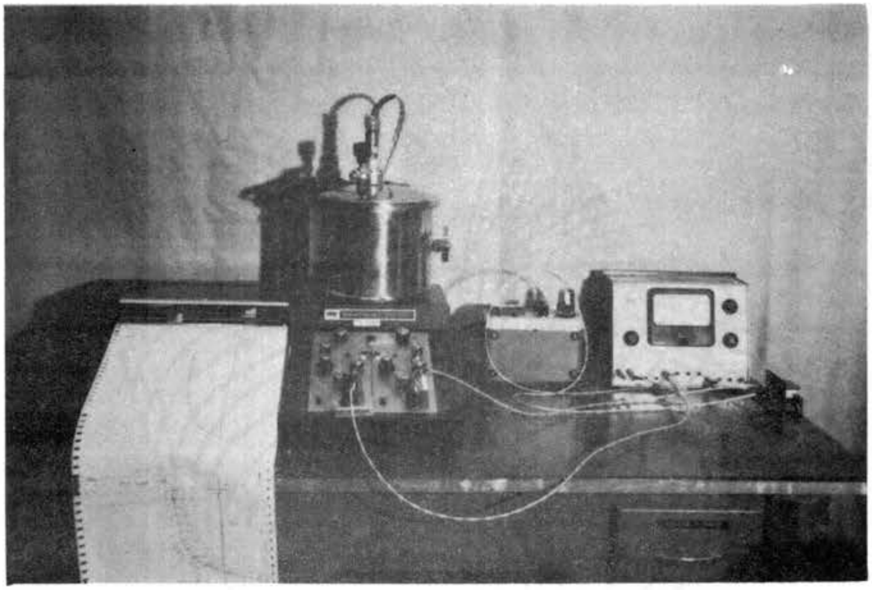


Figure 11a. View of Recorder, Tele-Thermometer and Pressure Container Device

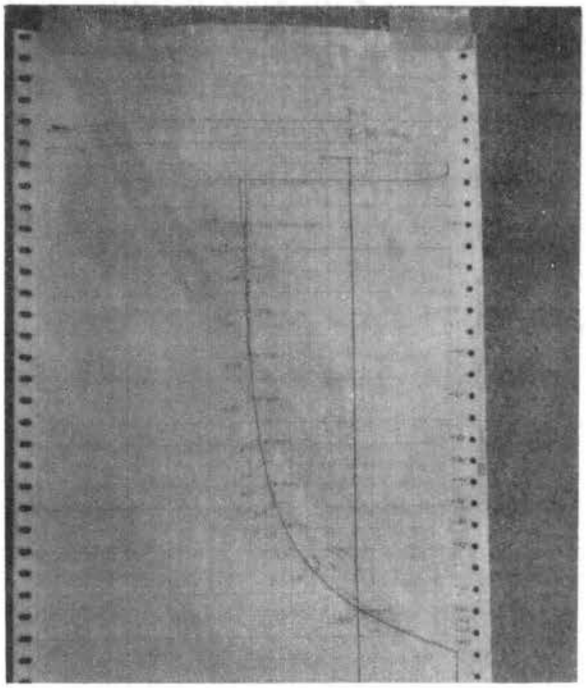


Figure 11b. View of Typical Strip-Chart Recorder Pressure Curve for Uncoated Limestone

on the chart paper for each 15 seconds of elasped reaction time. The pressure and temperature readings were scaled from the chart paper and tabulated. Pressures were adjusted to 68 F for comparative stripping calculations. This adjustment of pressures was necessary because of the slightly different operating temperatures and some reactions created higher temperatures than the standard 68 F.

The surface area exposed was taken as proportional to the change in pressure. For limestone aggregate mixtures, the change in pressure from 0.25 to 1.5 minutes of reaction time was assumed proportional to the exposed surface area. For siliceous aggregates, the change in gas pressure from 0.25 to 5.0 minutes of reaction time was assumed proportional to the exposed surface area. For aggregates of mixed composition, using mixtures of HCl and HF, the change in pressure taken from 0.25 to 1.5 minutes of reaction time was assumed proportional to the exposed surface area. The effect of inertia on the pressure transducer operation and the recorder chart pen response was the primary reason for using the initial gas pressure value at 0.25 minutes of reaction time. The reaction time used for the final pressure value was the time required to obtain a measurable pressure.

## Surface Area Related to Change in Gas Pressure

The hypothesis that change in gas pressure was proportional to exposed surface area was examined in the following manner. The Cooperton limestone aggregate was chosen to evaluate the surface area relationship with the change in gas pressure. Large pieces of rock, weighing from 5 to 10 lb, were obtained from the quarry adjacent to the working face. These rocks were sawed into slabs about 0.2 to 0.4 inches thick.

A diamond core drill, 0.75 inch inside diameter, was used to obtain discs of uniform diameter.

Each disc was numbered and its average thickness determined from five measurements using a micrometer dial gage. The device used is shown in Figure 5. The surface area of each disc was then calculated.

The disc was then divided into five different size groups of approximately 6000, 12,000, 18,000, 24,000 and 30,000 $mm^2$ each. Duplicate groups were prepared with approximately the same surface area.

Each group of discs was reacted with a HCl solution, using the test procedure previously outlined, and the resulting gas pressures evaluated. Results of this work will be presented later; however, it is noted that a linear relationship between change in gas pressure and limestone aggregate disc surface area was obtained.

## Reaction Between Asphalt Cement and Reagent

The hypothesis that asphalt cement will not react with the reagents used in the surface reaction test was investigated. Individual pieces of aggregate were partially imbedded in asphalt cement in a plastic container and then covered with the acid solution being evaluated. The reaction was observed visually and then later the specimen was examined microscopically using a magnification of 30X. Both HCl and HF solutions were evaluated with different types of rock and there were no visual effects noted of the acid solutions reaction with the asphalt cement.

To approximate the conditions encountered in the surface reaction test, duplicate asphalt cement coated aggregate samples were tested with the HCl solution and with the HF solution. The asphalt cement coated samples were prepared as described for sample preparation in the static stripping test, except 10 g of asphalt cement was used, rather than 6 g, to coat the 100 g aggregate sample. The coated aggregate sample was allowed to cool to about 140 F and then transferred to the plastic container used in the pressure vessel. After cooling the sample to room temperature, the surface reaction test was then performed on the asphalt cement coated sample. A small constant pressure (0.02 psi) was indicated when the HCl solution was reacted with the asphalt coated sample. The addition of the HF solution to its asphalt coated sample resulted in a small constant vacuum (0.03 psi) in the pressure vessel. These constant pressures did not affect the surface reaction test results since the surface area of the specimen were predicated on the change in gas pressure over a stated interval of time.

# CHAPTER V

## TEST RESULTS AND DISCUSSION

The test procedures described in Chapter IV were followed in performing stripping tests on the eleven aggregates under study. The stripping resistance of the aggregates were evaluated by the immersion-compression (I-C) test, static immersion stripping (SIS) test an dynamic immersion stripping (DIS) test.

The stripped aggregate samples from the dynamic immersion test and duplicate uncoated aggregate samples were tested in the surface reaction test to quantitatively evaluate the amount of stripping. Results of calibration tests relating the surface area of discs cut from Cooperton limestone and the gas pressure created from their reaction with dilute hydrochloric acid are also presented.

### Immersion-Compression Tests

Data are presented for I-C tests on specimens remolded at different densities. One series of specimens was remolded by the standard gyratory-shear procedure. Another series was remolded by a modified gyratory-shear procedure which yielded lower specimen densities. This method of molding was adopted for all I-C specimens and will be termed the modified procedure.

New specimens, representing each type of aggregate being studied were molded using the modified procedure. The I-C test was also performed on these specimens to determine their percentage retained strength. Data from these tests are presented to provide a relative indication of the stripping propensity exhibited by the different aggregates.

### Specimens Remolded by the Standard Procedure

The preliminary work with the immersion-compression testing method involved re-using specimens that had been batched, molded and tested for stability and cohesiometer values. The coarse aggregate (sized to pass the 3/4 in. sieve and be retained on the No. 10 sieve) used in these specimens were composed of Cooperton limestone in one series, and a blend of each aggregate under study and Cooperton limestone in the other mixtures. Only the coarse aggregate composition was varied in these trial specimens to obtain 20, 30, and 40 per cent by weight of acid insoluble material.

These specimens were reheated to 255 F, broken down by hand and remolded, using the standard compaction procedure. The asphalt cement content (by total weight of the mix) of the specimens ranged from 4.0 to 6.5 per cent. However, since the optimum asphalt content for these mixes was between 4.0 and 5.0 per cent, data will be present for only the 4.0, 4.5 and 5.0 per cent asphalt content specimens.

Test results are presented in Table VIII. The first series of tests were performed on the 20, 30, and 40 per cent acid insoluble mixes containing Asher chert gravel. Their index of retained strength ranged from 101 to 109 per cent, this is, all of the wet specimens were stronger than the dry specimens.

## TABLE VIII

### PHYSICAL PROPERTIES AND IMMERSION-COMPRESSION TEST RESULTS FOR MIXTURES REMOLDED BY STANDARD PROCEDURE

| Sample | AC (%) | Vacuum Saturated | Bulk Specific Gravity | Air Voids (%) | Dry Str. (psi) | Retained Strength (%) |
|---|---|---|---|---|---|---|
| Cooperton | 4 | yes | 2.399 | 4.2 | 477 | 103 |
| | 4 1/2 | yes | 2.413 | 2.9 | 425 | 102 |
| | 5 | yes | 2.429 | 1.6 | 316 | 105 |
| 20% Asher | 4 | no | 2.372 | 4.6 | 440 | 105 |
| | 4 1/2 | no | 2.386 | 2.8 | 378 | 108 |
| | 5 | no | 2.408 | 1.2 | 288 | 108 |
| 30% Asher | 4 | no | 2.355 | 4.3 | 425 | 109 |
| | 4 1/2 | no | 2.368 | 2.9 | 434 | 102 |
| | 5 | no | 2.388 | 1.4 | 322 | 108 |
| 40% Asher | 4 | no | 2.341 | 4.2 | 425 | 101 |
| | 4 1/2 | no | 2.356 | 2.7 | 426 | 103 |
| | 5 | no | 2.373 | 1.3 | 370 | 106 |
| 20% Miami | 4 | yes | 2.388 | 4.2 | 380 | 126 |
| | 4 1/2 | yes | 2.416 | 2.3 | 390 | 112 |
| | 5 | yes | 2.428 | 1.2 | 350 | 115 |
| 30% Miami | 4 | yes | 2.377 | 4.4 | 413 | 101 |
| | 4 1/2 | yes | 2.393 | 3.0 | 405 | 99 |
| | 5 | yes | 2.410 | 1.6 | 309 | 114 |
| 40% Miami | 4 | yes | 2.357 | 5.1 | 426 | 82 |
| | 4 1/2 | yes | 2.391 | 2.9 | 412 | 100 |
| | 5 | yes | 2.401 | 1.8 | 302 | 117 |

In an attempt to increase the severity of the test, a vacuum process was subsequently used to saturate the "wet" specimens. After vacuum saturation, the index of retained strength of the Cooperton limestone specimens ranged from 102 to 105 per cent. The 20, 30, and 40 per cent Miami chert aggregate specimens tested after vacuum saturation showed a scatter of retained strength from 82 to 126 per cent, with all but two tests being over 100 per cent.

The air void contents of the specimens ranged from 1.2 to 5.1 per cent. Considerable variation in percentage retained strength over relatively small ranges of void content was noted. Generally, however, the I-C retained strength increased as the voids in the specimen decreased. This decrease in air void content, of course, corresponds with the increase in asphalt content in the respective mixtures.

This preliminary work indicated that specimens with low air void contents would not exhibit a reduction in retained strength when compacted by the standard gyratory-shear procedures and tested by the modified I-C method used in this study. Generally, this was also true for specimens that were thoroughly soaked by the vacuum saturation process.

### Specimens Remolded by Modified Procedure

The amount of compactive effort applied during the molding process was reduced to obtain lower specimen densities, i.e., higher air void contents. This modified compaction procedure is discussed in detail in Chapter IV.

The same series of specimens previously remolded using standard compaction for the immersion-compression tests were heated, broken down and remolded using this modified procedure. The air void content in these twice remolded specimens ranged from 4.0 to 9.0 per cent. After remolding, the "wet" specimens from this series were subjected to vacuum saturation.

These results for this series of specimens are given in Table IX. As might be expected, the specimens containing 4 and 4.5 per cent asphalt contents and, consequently, larger amounts of voids showed some loss in retained strength. However, there were some exceptions in the various aggregate blends. Again, as in the previous series of specimens at asphalt contents near or above the optimum for the mixtures, per cent retained strengths above 100 were obtained.

Similar results were obtained on remolded specimens representing all of the eleven aggregate blends. No definite trends with regard to the relative stripping resistance of these blends could be established. Apparently, even when the amounts of siliceous aggregate approached 40 per cent by weight of the coarse aggregate in the specimen any detrimental effects on retained strength were too minor to influence the test results.

Good adhesion between asphalt and the surface of an aggregate particle is influenced to some extend by the length or time of contact, i.e., in many cases adhesion improves with time. This increase in adhesion may occur by molecular attraction, where the dipole molecules as asphalt orient themselves to satisfy the demands of polar aggregate. This molecular orientation proceeds slowly in a viscous liquid such as asphalt, but when achieved it will give a uniform adhesion to the aggregate surface.

## TABLE IX

## PHYSICAL PROPERTIES AND IMMERSION-COMPRESSION TEST RESULTS FOR MIXTURES REMOLDED BY MODIFIED PROCEDURE

| Sample | AC (%) | Water Absorbed % (vacuum sat.) | Bulk Specific Gravity | Air Voids (%) | Dry Str. (psi) | Retained Strength (%) |
|---|---|---|---|---|---|---|
| Cooperton | 4 | 3.6 | 2.300 | 8.7 | 322 | 86 |
| | 4 1/2 | 3.0 | 2.323 | 7.1 | 312 | 92 |
| | 5 | 2.1 | 2.351 | 5.2 | 319 | 96 |
| 20% Asher | 4 | 3.8 | 2.270 | 8.4 | 407 | 76 |
| | 4 1/2 | 2.8 | 2.300 | 6.3 | 380 | 101 |
| | 5 | 1.7 | 2.399 | 4.0 | 356 | 104 |
| 30% Asher | 4 | 3.6 | 2.270 | 7.8 | 409 | 84 |
| | 4 1/2 | 2.9 | 2.288 | 6.2 | 402 | 102 |
| | 5 | 1.9 | 2.326 | 4.0 | 395 | 106 |
| 40% Asher | 4 | 4.0 | 2.244 | 8.1 | 336 | 96 |
| | 4 1/2 | 3.3 | 2.269 | 6.2 | 375 | 98 |
| | 5 | 2.5 | 2.294 | 4.5 | 294 | 100 |
| 20% Miami | 4 | 4.2 | 2.268 | 9.0 | 340 | 96 |
| | 4 1/2 | 3.0 | 2.305 | 6.8 | 320 | 106 |
| | 5 | 2.3 | 2.326 | 5.3 | 329 | 110 |
| 30% Miami | 4 | 3.8 | 2.282 | 8.2 | 381 | 85 |
| | 4 1/2 | 3.2 | 2.295 | 7.0 | 370 | 95 |
| | 5 | 2.2 | 2.325 | 5.1 | 304 | 102 |
| 40% Miami | 4 | 4.1 | 2.268 | 8.7 | 312 | 88 |
| | 4 1/2 | 2.9 | 2.301 | 6.6 | 332 | 93 |
| | 5 | 2.3 | 2.321 | 5.1 | 273 | 104 |

Hardness of the asphalt film is also a factor. The harder the asphalt film, provided the asphalt had attained good adhesion, the more difficult it is for water to displace it. The data presented in Table II may be used to illustrate the effect of hardness (low viscosity) on stripping resistance of two different penetration grade asphalt cements. The quartzite aggregate increased its retained coating in a DIS test from 53 to 75 per cent when the penetration of the asphalt cement was decreased from 85-100 to 50-60 penetration. The limestone aggregate also indicated an increase in retained coating from 95 to 99 per cent when the harder asphalt was used.

Continued heating at high temperatures may also increase the hardness of the asphalt cement. This heating tends to drive off the more volatile of the oils in the asphalt and causes oxidation which contributes to hardness and the development of insoluble matter in the asphalt (67). This hardening effect on the asphalt cement used in this work may be illustrated by examinating physical properties of the asphalt cement which were presented in Table IV. The asphalt cement was subjected to the thin film oven (TFO) test, in which a thin layer of asphalt (about 1/8 in. thick) was heated for 5 hours at 325 F. The penetration of the asphalt decreased from 93 to 60 in this period of time. This loss of penetration should be indicative of the hardening effect that would take place in the mixing process of asphalt-aggregate mixtures where a thin film of asphalt surrounds aggregate at temperatures similar to those in the TFO test.

This possible effect of curing temperature increasing the hardness (and therefore the strength) of the asphalt cement in the I-C "wet" specimens was investigated in this preliminary work. It was thought that the wet specimens were stronger than the dry specimens because of their 24 hour immersion in a 140 F water bath. One series of I-C test were performed where the dry specimens were cured at 140 F in an oven during the same time as the wet specimens were being soaked at 140 F. Retained strength test results on these I-C specimens were similar to the data presented in Table VIII and Table IX. It was concluded that no measurable hardening of the asphalt occurs as a result of the 24 hour immersion of the wet specimen in the 140 F water bath.

The re-use of previously molded specimens (some two or three months old) permitted long contact times between the asphalt and aggregate surfaces. The remolding procedure resulted in re-heating the mixtures and re-mixing, thus tending to harden the asphalt. This re-heating of the asphalt while in contact with the aggregate would possibly give the dipole molecules in the asphalt cement an opportunity to properly orient themselves for maximum adhesion to siliceous aggregates.

For these reasons the use of old specimens and the remolding process may have been responsible for the lack of good conclusive information. However, aging and cycles of re-heating and manipulation under traffic are also processes undergone in an actual pavement. Therefore, these results are probably more or less truly indicative of the responses of these mixtures under field conditions.

### Specimen Molded Primarily for I-C by Modified Procedure

The previously discussed I-C results failed to yield definite indications of the stripping tendencies of the respective aggregate blends. Therefore, mixtures for each of the eleven aggregate types were prepared separately, and compacted by the modified gyratory-shear procedure.

The coarse aggregate portion (plus No. 10 material) of the mixtures was composed entirely of the respective aggregate to be evaluated. The fine aggregate fraction (minus No. 10 material) was held constant for each specimen, i.e., the fine aggregate materials were identical for each of these mixtures. Thereby, effects of the fine aggregate on the immersion-compression retained strengths should be the same for each specimen. This fine aggregate was composed of a high quality sand from the Arkansas River, with the mineral filler being obtained from screenings of the Cooperton limestone. For each of the eleven aggregates, four specimens were molded at 4 and 5 per cent asphalt content, respectively.

Table X shows the physical properties of these specimens and the results of the I-C tests. The Cooperton limestone specimens (5 per cent asphalt content) had the highest retained strength at 116 per cent, while the lowest retained strength of 66 per cent was evidenced by the Onapa sandstone mixture (4 per cent asphalt content). It is observed that, at 4 per cent asphalt contents, the limestone aggregates (Cooperton, Hartshorne and Stringtown) averaged 103 per cent retained strength, while the sandstone aggregates (Cyril, Keota and Onapa) average value was 78 per cent retained strength. Likewise, the Miami chert and the gravels (Asher, Broken Bow, Gore and Hugo) had an average retained strength of 91 per cent.

Based on the aggregate classification system shown in Figure 1, it is noted that the electropositive (limestones) aggregates are at one end of the classification scale while the electronegative (sandstones)

# TABLE X

## PHYSICAL PROPERTIES AND IMMERSION-COMPRESSION TEST RESULTS FOR MIXTURES MOLDED PRIMARILY FOR THE IMMERSION-COMPRESSION TEST, BY THE MODIFIED PROCEDURE

| Sample | AC (%) | Water Absorbed % (vacuum sat.) | Bulk Specific Gravity | Air Voids (%) | Dry Str. (psi) | Retained Strength (%) |
|---|---|---|---|---|---|---|
| Cooperton | 4 | 3.1 | 2.326 | 6.9 | 203 | 114 |
| | 5 | 1.5 | 2.375 | 3.5 | 214 | 116 |
| Hartshorne | 4 | 3.2 | 2.315 | 7.2 | 208 | 101 |
| | 5 | 1.6 | 2.360 | 3.8 | 228 | 104 |
| Stringtown | 4 | 2.3 | 2.249 | 7.8 | 246 | 95 |
| | 5 | 2.2 | 2.289 | 4.7 | 248 | 102 |
| Cyril | 4 | 3.5 | 2.298 | 7.4 | 251 | 88 |
| | 5 | 1.7 | 2.346 | 4.0 | 248 | 106 |
| Keota | 4 | 2.4 | 2.192 | 8.3 | 226 | 80 |
| | 5 | 2.3 | 2.248 | 4.6 | 236 | 97 |
| Onapa | 4 | 5.6 | 2.144 | 9.2 | 250 | 66 |
| | 5 | 2.7 | 2.194 | 5.8 | 290 | 76 |
| Asher | 4 | 3.6 | 2.189 | 7.8 | 249 | 95 |
| | 5 | 1.7 | 2.237 | 4.4 | 252 | 104 |
| Broken Bow | 4 | 3.1 | 2.286 | 6.4 | 233 | 94 |
| | 5 | 1.4 | 2.337 | 3.0 | 238 | 106 |
| Gore | 4 | 2.3 | 2.238 | 6.9 | 250 | 80 |
| | 5 | 1.5 | 2.282 | 3.7 | 254 | 96 |
| Hugo | 4 | 2.2 | 2.266 | 7.0 | 182 | 99 |
| | 5 | 2.2 | 2.324 | 3.2 | 186 | 105 |
| Miami | 4 | 3.0 | 2.244 | 8.1 | 235 | 89 |
| | 5 | 2.2 | 2.290 | 4.8 | 241 | 101 |

aggregates are placed at the other end of the scale. Using the averaged I-C test results, it would be concluded that, in general, the electropositive aggregates are more resistant to the stripping effects of water than the electronegative aggregates. However, the gravel aggregates contain (reference Table VI) as much or more acid insoluble materials "$SiO_2$" than do the sandstone aggregates and based on the criteria of Table I they would be classified as a more electronegative aggregate. Since the I-C retained strengths of the gravels were greater than the sandstones, it is seen that an aggregates stripping resistance is not necessarily related to its chemical composition.

For each of the aggregate mixtures there was an increase in retained strength with an increase in asphalt content. Further, most of the mixtures indicated greater than 100 per cent retained strength at 5 per cent asphalt content. The reason for this increase in strength, after soaking for 24 hours in a 140 F water bath, has not been fully explained. Other researchers have observed similar results (15, 47, 51, 53). The larger asphalt cement contents result in smaller air voids in the compacted specimens and these voids may be connected by extremely small capillaries. The entry of water into the capillaries, without causing stripping of the asphalt cement, creates a tensile force between particles of coated aggregate which results in a higher indicated ultimate strength of the specimen (53).

A similar phenomenon has been observed in tests of saturated soil specimens (68). The cause of an increase in the indicated strength of the specimen is attributed to negative porewater pressure. The increase in strength occurs in a dense, saturated specimen having small capillaries. Under a compressive load, if the specimen tends to swell or expand its volume, the water contained by the small capillaries resist this increase in volume. This negative pore-water pressure has the same effect as an increase in confining pressure on the specimen and results in a higher indicated ultimate strength.

The effects of air void content on I-C strength for this series of test are shown in Figure 12. The overall trend is for an increase in the I-C retained strength with decreasing air voids. The best fitted linear relationship lines were plotted for the 4 and 5 per cent asphalt specimens. The best fitted line was obtained by regression analysis, using a standard computer program. The best fitted equation for the 4 per cent asphalt specimens was:

$$Y = 171 - 10.6\ X$$

where: $X$ = air voids in mix in per cent

$Y$ = I-C retained strength in percent.

The coefficient of correlation between the data points and the best fitted equation was 0.666. Using the same type equation for the 5 per cent asphalt specimens, the best fitted equation was:

$$Y = 139 - 9.03\ X$$

The coefficient of correlation was 0.749 for the linear relationship between per cent air voids in the specimen and the immersion-compression retained strength.

## Summary of Immersion-Compression Results

The I-C retained strengths for each group of specimens prepared using the various aggregates have been reported. Factors thought to have affected these results were discussed. These factors included:

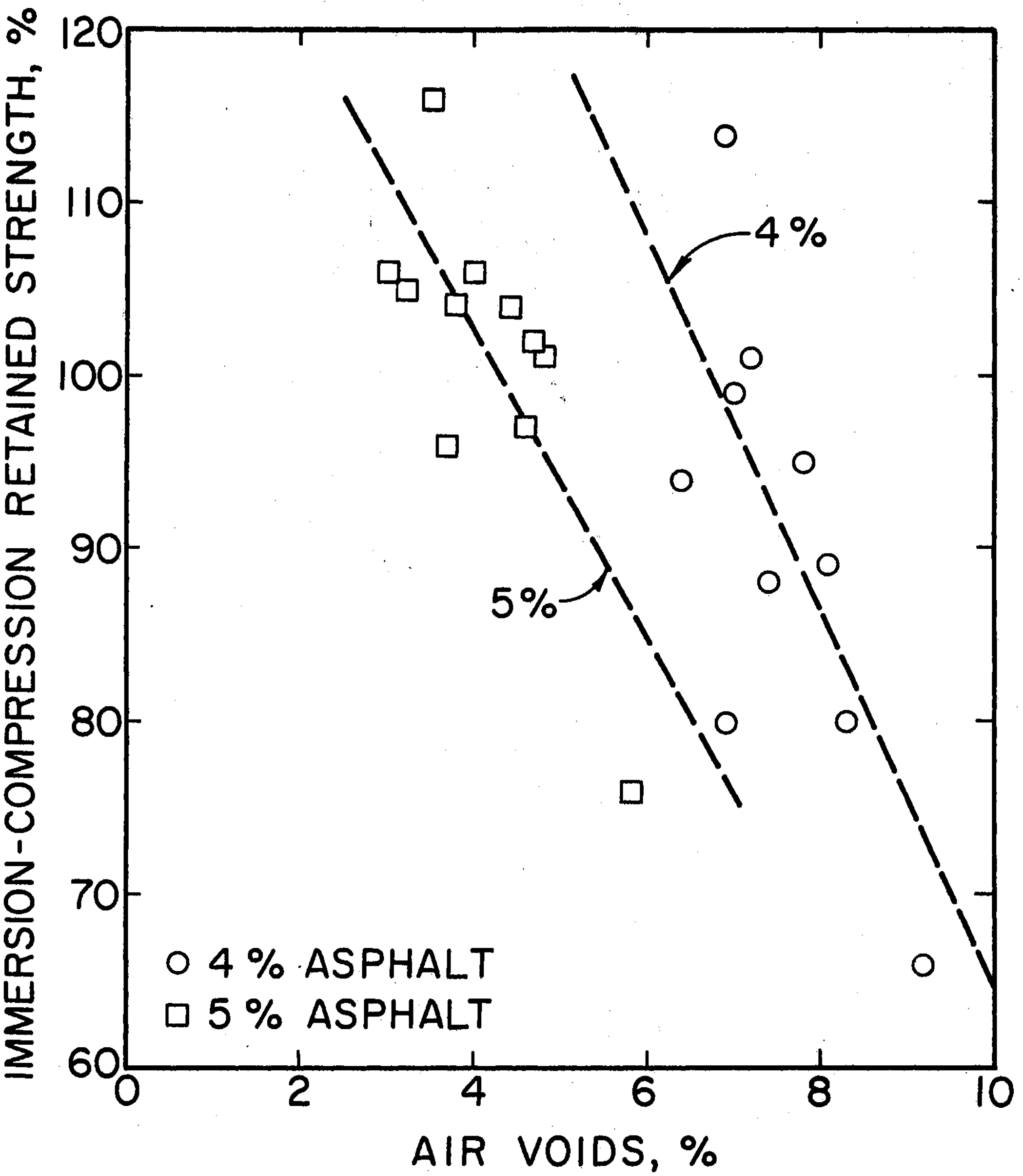


Figure 12. Percent Air Voids Versus Immersion-Compression Retained Strength for Specimens Molded Primarily for I-C, Modified Procedure

1) Method of compacting specimen.

2) Percentage of asphalt cement in the mixture.

3) Amount of air voids in the compacted specimen.

4) Effects of using remolded specimens.

5) Effects of temperature and aging on the hardening and adhesion characteristics of the asphalt.

6) Chemical composition of the aggregate.

For the purposes of this research it was desired to ascertain the relative stripping tendencies of the various aggregates under study, based on their immersion-compression retained strengths. Therefore, based on the I-C retained strength data presented in Table X, the relative stripping resistance of the aggregates (from excellent to poor) is: 1) Cooperton limestone, 2) Hartshorne limestone, 3) Hugo chert gravel, 4) Asher chert gravel, 5) Stringtown siliceous limestone, 6) Broken Bow siliceous gravel, 7) Miami chert, 8) Cyril calcareous sandstone, 9) Keota siliceous sandstone, 10) Gore siliceous gravel, and 11) Onapa siliceous sandstone.

## Static Immersion Stripping Tests

Samples of the various aggregates, being evaluated in this study, were prepared in 100 g groups after sieving, washing, drying, quartering and weighing as described in detail in Chapter IV. For the static immersion stripping tests the aggregate sample and asphalt cement were heated to 250 F prior to mixing together. Duplicate samples of each aggregate type were coated with approximately 6 g of asphalt cement.

After cooling to room temperature, the coated aggregate sample was placed in a glass jar and covered with 600 ml of distilled water.

The pH of this distilled water was about 5.4. The jar was capped and left undisturbed for 18 hours in a 77 F water bath. Visual estimation of the amount of stripping that occurred at the end of the 18 hours revealed no stripping of the asphalt on any of samples.

The glass jars, containing the coated aggregate sample, were then placed in a 140 F water bath for 18 hours of additional immersion time. Visual estimation of the amount of stripping was determined at the end of this second 18 hour immersion period using the comparison chart of Figure 7.

The comparison chart was used in the following manner to estimate the percentage area coated on the samples. The chart was placed flat on the table top adjacent to the jar sample to be evaluated. The top was removed from the jar and any film of asphalt on the surface of the water was removed by skimming with a paper towel. The jar was then placed adjacent to the chart and by visual observation of the sample, from above through the water, the jar was moved until the uncovered area of the comparison chart was matched by the sample. It was difficult to estimate the area covered with asphalt any closer than to the nearest 5 per cent. Therefore, duplicate test results were averaged and then rounded off to the nearest 5 per cent.

For the SIS test at 140 F, an estimated retained coating of 95 per cent on the Hugo chert gravel aggregate sample was the maximum value obtained. Other aggregate had more stripping evident, with the asphalt cement tending to recede from the sharper edges of the aggregate where the film was thinnest. The Gore siliceous gravel was then least coated sample with an estimated 60 per cent of its surface area exposed.

It is noted that these 22 SIS test specimens were prepared consecutively and tested as a group at one time. All evaluations were performed in a like manner at the same time. Average results of these 22 static immersion stripping tests, both at 77 F and 140 F, are reported in Table XI.

## Dynamic Immersion Stripping Tests

Asphalt coated samples were prepared for each aggregate, in duplicate, similarly to the procedure described for the static immersion stripping test. The sample, after coating, was placed in cold water to reduce its tendency to stick together prior to initiating the test. The duplicate samples were transferred to glass jars, covered with 600 ml of distilled water and tightly capped. The jars were placed in the DIS testing machine and tumbled for 1 hour, after which they were removed from the machine and the amount of surface area coated with asphalt visually estimated using the comparison chart (Figure 7) and method previously described. This testing and evaluating procedure was repeated for 2 hours and 4 hours of tumbling action. Retained asphalt coatings for each aggregate sample after 1, 2, and 4 hours of DIS testing are reported in Table XI.

After 1 hour of tumbling, all aggregate samples retained 90 per cent or more of their original asphalt coating and at the end of 2 hours of tumbling most of the samples still retained more than 85 per cent of their asphalt coating. After 4 hours of tumbling in the DIS test, the water in each jar became murky and was replaced with fresh water prior to visual evaluation. The turbid water resulted from loosened particles of asphalt cement and small particles that abraded from the aggregate

## TABLE XI

## RESULTS OF STATIC IMMERSION AND DYNAMIC IMMERSION STRIPPING TESTS

| Aggregate | Static Immersion Ret. Coating (%) | | Dynamic Immersion Ret. Coating (%) | | |
|---|---|---|---|---|---|
| | 77 F | 140 F | 1 hr. | 2 hr. | 4 hr. |
| Cooperton | 100 | 85 | 95 | 90 | 85 |
| Hartshorne | 100 | 75 | 95 | 90 | 75 |
| Stringtown | 100 | 65 | 95 | 90 | 85 |
| Cyril | 100 | 60 | 90 | 80 | 75 |
| Keota | 100 | 50 | 95 | 90 | 80 |
| Onapa | 100 | 50 | 95 | 90 | 85 |
| Asher | 100 | 90 | 95 | 90 | 80 |
| Broken Bow | 100 | 90 | 95 | 90 | 70 |
| Gore | 100 | 40 | 90 | 85 | 65 |
| Hugo | 100 | 95 | 95 | 90 | 80 |
| Miami | 100 | 70 | 95 | 85 | 75 |

surfaces. The values of retained coating ranged from 85 per cent for the Cooperton limestone, Keota sandstone and Onapa sandstone to 65 per cent for the Gore siliceous gravel.

The samples were removed from the glass jars after their 4 hour evaluation and air dried, in preparation for their surface reaction test. In general, two different types of aggregates were subjected to the DIS test at the same time.

A comparison of the various aggregate retained coatings (Table XI) for the SIS and DIS test results show little correlation. Mathews, et al. (48) also reported little correlation of test results between the static immersion and dynamic immersion tests performed on 16 different types of aggregates. Factors affecting the visual estimation of retained coatings will be discussed later.

## Surface Reaction Tests

A quantitative evaluation of the amount of stripping (due to the action of water) undergone by asphalt cement coated aggregate particles was the purpose of this reaearch. The surface reaction test (SRT) was developed to accomplish this purpose.

Several methods for surface area measurement are briefly discussed. Surface area-gas pressure calibration relationships for Cooperton limestone disc were obtained and asphalt cement coated samples, partially stripped in the dynamic immersion stripping test, were evaluated by the surface reaction test.

## Surface Area Determinations

The irregular shape, varying surface texture and pore size of most aggregate particles are infinite in number and for these reasons no accurate determination of the aggregate surface area is possible at the present time. However, the surface area of solid particles may be estimated very closely by several methods.

Fries (69) reported several adsorption methods of estimating surface area for very finely divided particles. These adsorption methods are based on determining the amount of gas or liquid, at a constant pressure and temperature, required to just cover the surface of the solid with a monomolecular layer. The surface can then be evaluated if the area occupied by each molecule in this monolayer is known.

Gas permeability is another way of estimating surface area, and utilizes the resistance to flow of air or a fluid through a mass of powder. The specific surface of the powder is calculated by a semi-empirical equation. Fries (69) also presented data from the literature showing a comparison of surface area determination by two different methods. For a material composed of glass spheres, the surface areas were: 1) by nitrogen adsorption, area = 0.55 $m^2/g$, and 2) by gas permeability, area = 0.287 $m^2/g$. The surface area differences between these two methods were attributed to the porous structure of the glass spheres.

In 1918, Edwards (70) proposed a method of estimating the surface area of aggregate particles which appeared to give comparative results. The aggregate was separated into various component sizes and the number of particles per unit weight of each size was obtained by count. The bulk specific gravity of each component size was determined and the solid volume of each particle was calculated. The shape of the particles was assumed as spherical and the surface area per particle was calculated. The total surface area of an aggregate mixture could then be determined.

Hatt and Scofield (71), using Edward's method, developed the following formula to calculate the surface area of any size of aggregate separation.

$$A = 48,313\ (N/S^2)^{1/3}$$

where: A = surface area in $mm^2$/100 g

N = number of particles per g

S = bulk specific gravity

Surface Area and Specific Gravity of Aggregate Samples. The surface area of each 100 g aggregate sample was determined using Hatt and Scofields formula. The average surface areas for each type aggregate used in the surface reaction test are given in Table XII. The average specific gravity and absorption for each aggregate type, along with the average number of aggregate particles counted for the 100 g samples are also reported in Table XII.

SRT Calibration Test Results. To determine the relationship between aggregate surface area and gas pressure created, calibration tests for the Cooperton limestone were performed. Uniform discs were obtained from the Cooperton limestone rocks by sawing the rocks into slabs about 0.3 inch thick and then drilling disc with a 0.75 inch diameter diamond core drill. The size of each disc was measured and its surface area then calculated. Five sets of discs, with known surface areas ranging

## TABLE XII

## SURFACE AREA AND SPECIFIC GRAVITIES OF 100 GRAM SAMPLES

| Aggregate | Average Number Particles per 100 g. | Bulk[1] Specific Gravity | Apparent[1] Specific Gravity | Absorption[1] (%) | Surface Area ($mm^2/100g$) |
|---|---|---|---|---|---|
| Cooperton | 115 | 2.684 | 2.717 | 0.5 | 26,180 |
| Hartshorne | 133.8 | 2.651 | 2.706 | 0.8 | 27,725 |
| Stringtown | 149.5 | 2.535 | 2.608 | 1.1 | 29,680 |
| Cyril | 140 | 2.590 | 2.697 | 1.5 | 28,625 |
| Keota | 151 | 2.405 | 2.611 | 3.3 | 30,850 |
| Onapa | 173.2 | 2.323 | 2.607 | 4.7 | 33,050 |
| Asher | 143 | 2.376 | 2.562 | 3.1 | 30,550 |
| Broken Bow | 134.8 | 2.534 | 2.631 | 1.5 | 28,680 |
| Gore | 145.8 | 2.432 | 2.594 | 2.5 | 30,180 |
| Hugo | 167 | 2.510 | 2.606 | 1.4 | 31,000 |
| Miami | 154.2 | 2.522 | 2.630 | 1.6 | 30,050 |

[1] Reference ASTM Designation: C127.

from 6000 $mm^2$ to 30,000 $mm^2$, were subjected to the surface reaction test. Duplicate groups, with approximately the same surface area, were tested. The gas pressure resulting from the reaction of these Cooperton limestone discs and dilute hydrochloric acid was measured.

The surface area of each disc sample with the corresponding increase in gas pressure for a reaction time of from 15 to 90 seconds is shown in Figure 13. The initial gas pressure reading at 15 seconds was used to reduce any lag or inertia effects of the recording equipment. The final pressure reading was arbitrarily chosen at 90 seconds, such that all of the exposed aggregate surface had only been slightly etched by the acid solution. A fairly good linear relationship between disc surface area and change in gas pressure is noted. The increase in gas pressure was approximately 0.059 psi for each 1000 $mm^2$ of disc surface area. While time did not permit the "calibration" of other aggregates used in this study, a similar relationship of surface area to gas pressure increase would be anticipated.

The appearance of the discs "before" the calibration test are shown in the photograph of Figure 14. Each disc had a surface area of about 1000 $mm^2$. The reaction between the acid and the discs was stopped after 6 minutes of reaction time. The discs were then washed, oven dried and their new diameter determined by measurement. During the test the discs turned a darker color and veins of calcite became evident on some of them. This can be seen in the "after" photograph of Figure 15.

The relationship between original surface area of the discs and the thickness of material etched or dissolved during the 6 minutes of reaction time is shown in Figure 16. For a disc sample whose surface

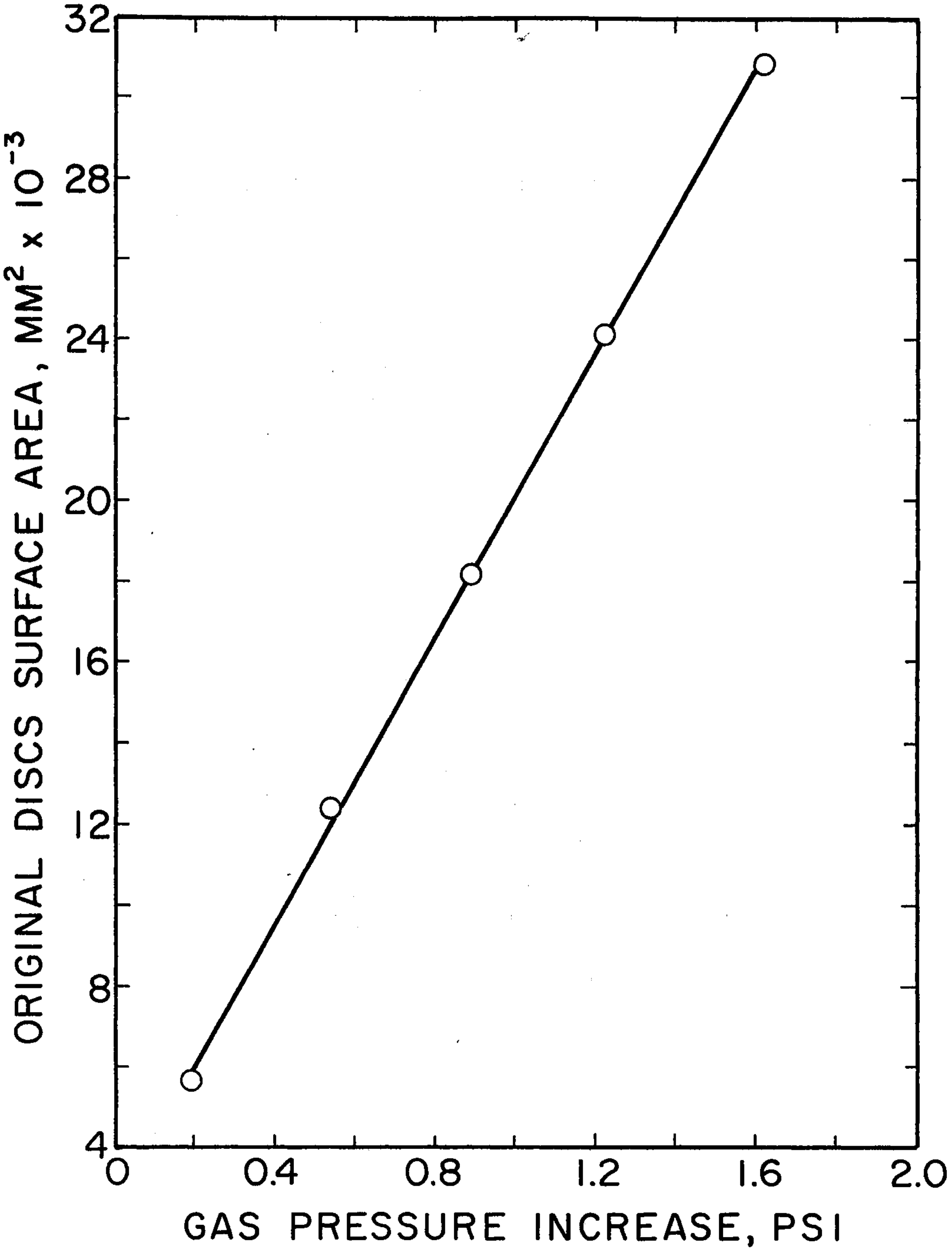


Figure 13. Original Disc Surface Area Versus Gas Pressure Increase, Cooperton Limestone Discs

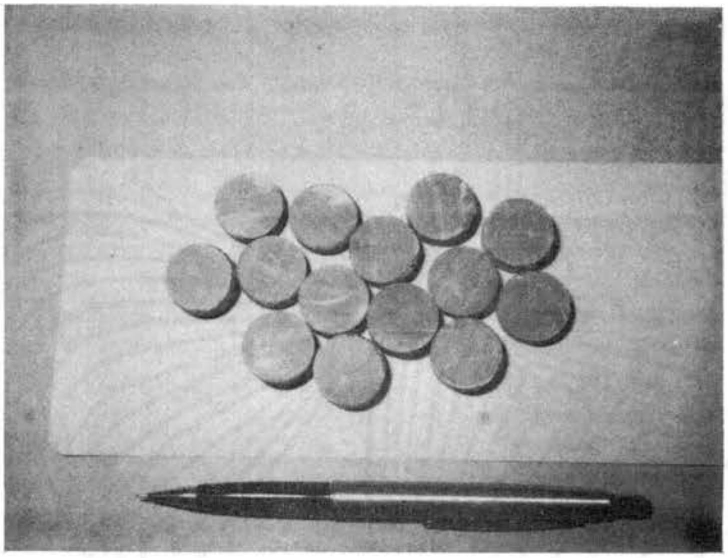


Figure 14. Cooperton Limestone Disc Before Surface Reaction Calibration Test

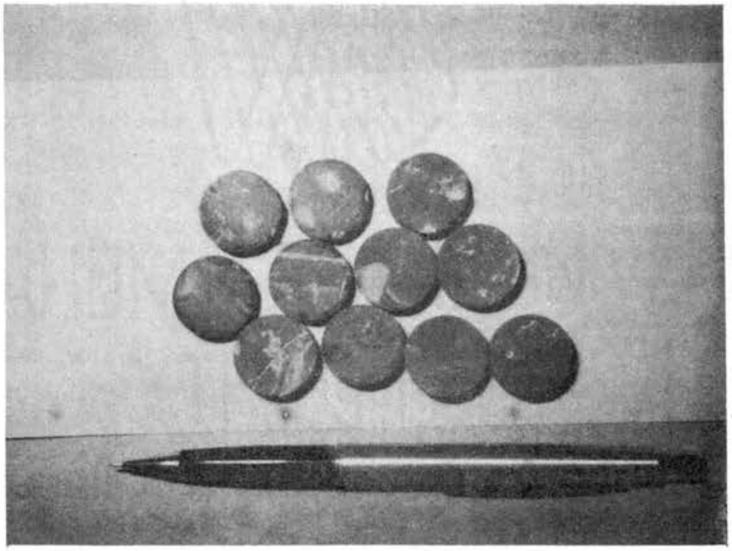


Figure 15. Cooperton Limestone Disc After Surface Reaction Calibration Test

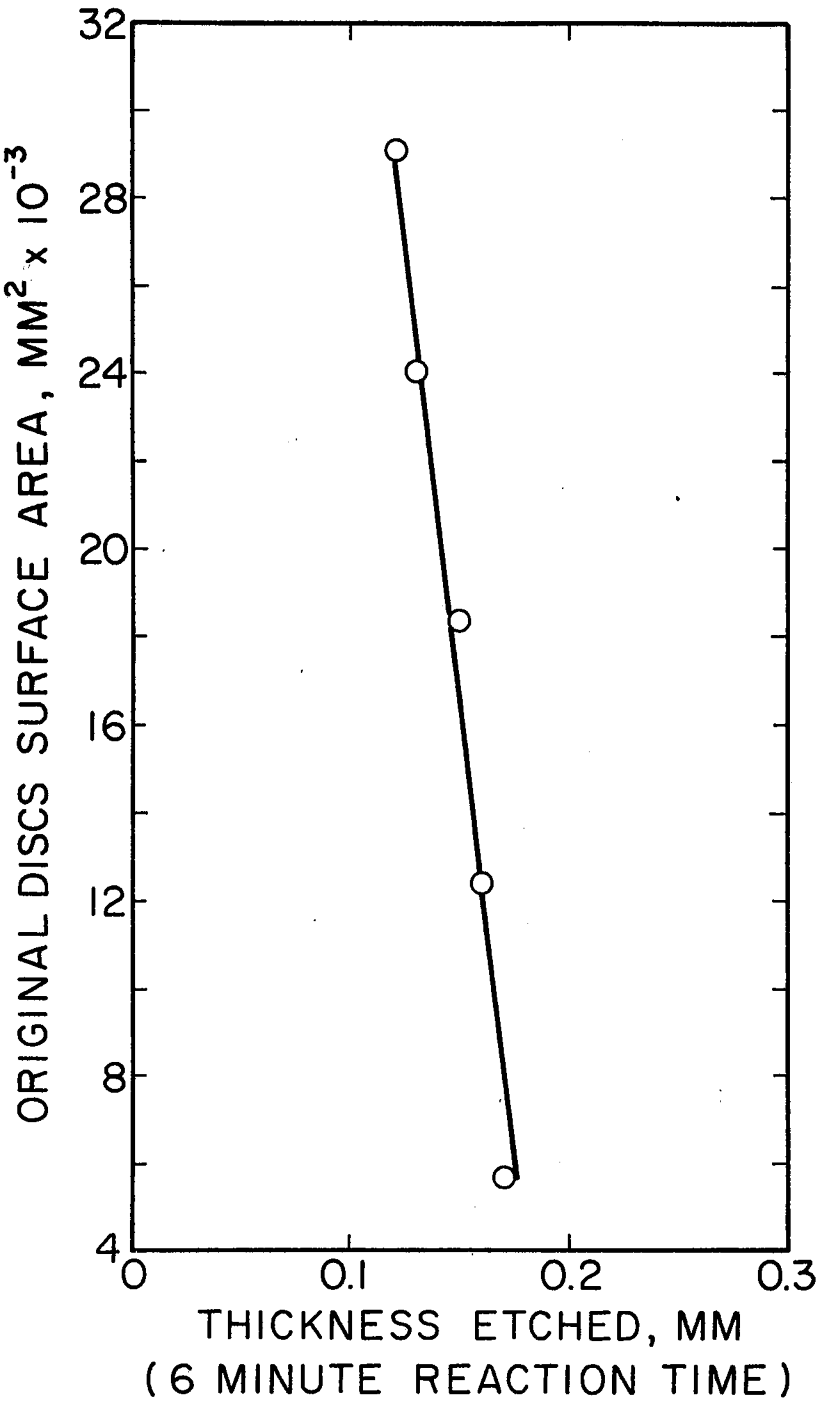


Figure 16. Original Disc Surface Area Versus Thickness Etched, Cooperton Limestone Discs

area was 28,000 $mm^2$, the material was etched off at the rate of 0.026 mm per minute. With a disc sample area of 8,000 $mm^2$, the material was etched off at the rate of 0.028 mm per minute.

## Surface Reaction Test Results

The surface reaction test was performed on each aggregate under study. The test procedure involved placing the 100 g sample to be evaluated in the pressure vessel, adding 200 ml of acid solution and obtaining the resulting pressure and temperature on a strip-chart recorder. The detailed procedure was given in Chapter IV.

Each aggregate was tested both uncoated and partially coated with asphalt. Duplicate samples were used in all cases, the partially coated samples were obtained from the dynamic immersion stripping tests. The test pressures obtained were adjusted to 68 F. This was necessary because the test temperatures varied slightly from test to test and also some mixtures created higher gas temperatures. Duplicate test values were in close agreement with one another, differing less than 0.02 psi in most cases, and were averaged for both the uncoated and partially coated samples. These average values from the SRT performed on each aggregate were used in the analysis of the test results.

The temperature adjusted pressure-time curve obtained from the strip-chart recorder was analyzed to obtain the best fitted equation. This was accomplished by regression analysis using a standard computer program. The pressure-time curve plotted approximately as a straight line on log-log paper and was a parabola or hyperbola of the form $Y = CX^B$. It is convenient to express this equation in the form:

$$\log Y = \log C \pm B \log X$$

where: X = time in minutes

Y = pressure in psi

B = slope of the straight line

C = value of Y when X = 1.0, i.e., the intercept of the straight line.

The coefficients of this best fitted equation along with its coefficient of correlation (R) for both uncoated (U) and partially stripped (S) samples were determined for each aggregate tested and are presented in Table XIII.

A typical strip-chart recorder trace of the pressures obtained for an uncoated limestone aggregate sample and a partially coated limestone aggregate sample are shown in Figure 17. The initial pressure reading was taken at 15 seconds of reaction time and is shown as $P_1$, while the final pressure reading was taken at 90 seconds of reaction time and is shown as $P_2$, in Figure 17. The retained coating of asphalt is calculated as follows:

$$RC = 100 - \left(\frac{\Delta P_s}{\Delta P_u}\right) 100$$

where: RC = retained coating in percent

$\Delta P_s = P_2 - P_1$ for the stripped sample

$\Delta P_u = P_2 - P_1$ for the uncoated sample.

For example, using the values shown in Table XIII for the Cooperton limestone, $\Delta P_s$ = 0.20 psi, $\Delta P_u$ = 2.10 psi, then:

$$RC = 100 - \left(\frac{0.20}{2.10}\right) 100$$

$$RC = 100 - 9.52$$

RC = 90 per cent.

## TABLE XIII

## SURFACE REACTION TEST RESULTS

| Aggregate | Surface[1] Condition | Coefficients Best Fitted Equation | | Corr. Coef. | ΔP | Retained Coating | |
|---|---|---|---|---|---|---|---|
| | | C | B | R | | (%) | Range[2] |
| Cooperton | S | -0.739 | 0.909 | 0.997 | 0.20 | 90 | ±0.5 |
| | U | 0.250 | 0.903 | 0.996 | 2.10 | | |
| Hartshorne | S | -0.582 | 0.831 | 0.995 | 0.28 | 85 | ±0.5 |
| | U | 0.214 | 0.875 | 0.998 | 1.88 | | |
| Stringtown | S | -0.918 | 0.877 | 0.987 | 0.14 | 93 | ±0.7 |
| | U | 0.312 | 0.766 | 1.000 | 2.08 | | |
| Cyril | S | -1.210 | 0.530 | 0.995 | 0.05 | 64 | ±0.7 |
| | U | -0.765 | 0.595 | 0.996 | 0.14 | | |
| Keota | S | -1.49 | 0.595 | 0.993 | 0.07 | 56 | ±0 |
| | U | -1.24 | 0.707 | 0.998 | 0.16 | | |
| Onapa | S | -1.61 | 0.656 | 0.998 | 0.06 | 68 | ±0.8 |
| | U | -1.27 | 0.825 | 0.993 | 0.19 | | |
| Asher | S | -1.40 | 0.598 | 0.983 | 0.08 | 74 | ±3.4 |
| | U | -1.20 | 0.958 | 0.995 | 0.31 | | |
| Broken Bow | S | -1.42 | 0.503 | 0.993 | 0.06 | 54 | ±3.6 |
| | U | -1.16 | 0.480 | 0.992 | 0.13 | | |
| Gore | S | -1.43 | 0.598 | 0.994 | 0.08 | 65 | ±2.2 |
| | U | -1.20 | 0.816 | 0.998 | 0.23 | | |
| Hugo | S | -1.40 | 0.461 | 0.981 | 0.07 | 78 | ±0.5 |
| | U | -1.27 | 0.984 | 0.998 | 0.27 | | |
| Miami | S | -1.39 | 0.549 | 0.992 | 0.08 | 60 | ±3.5 |
| | U | -1.13 | 0.626 | 0.992 | 0.20 | | |

[1] S = partially stripped, U = uncoated.

[2] Range based on duplicate tests.

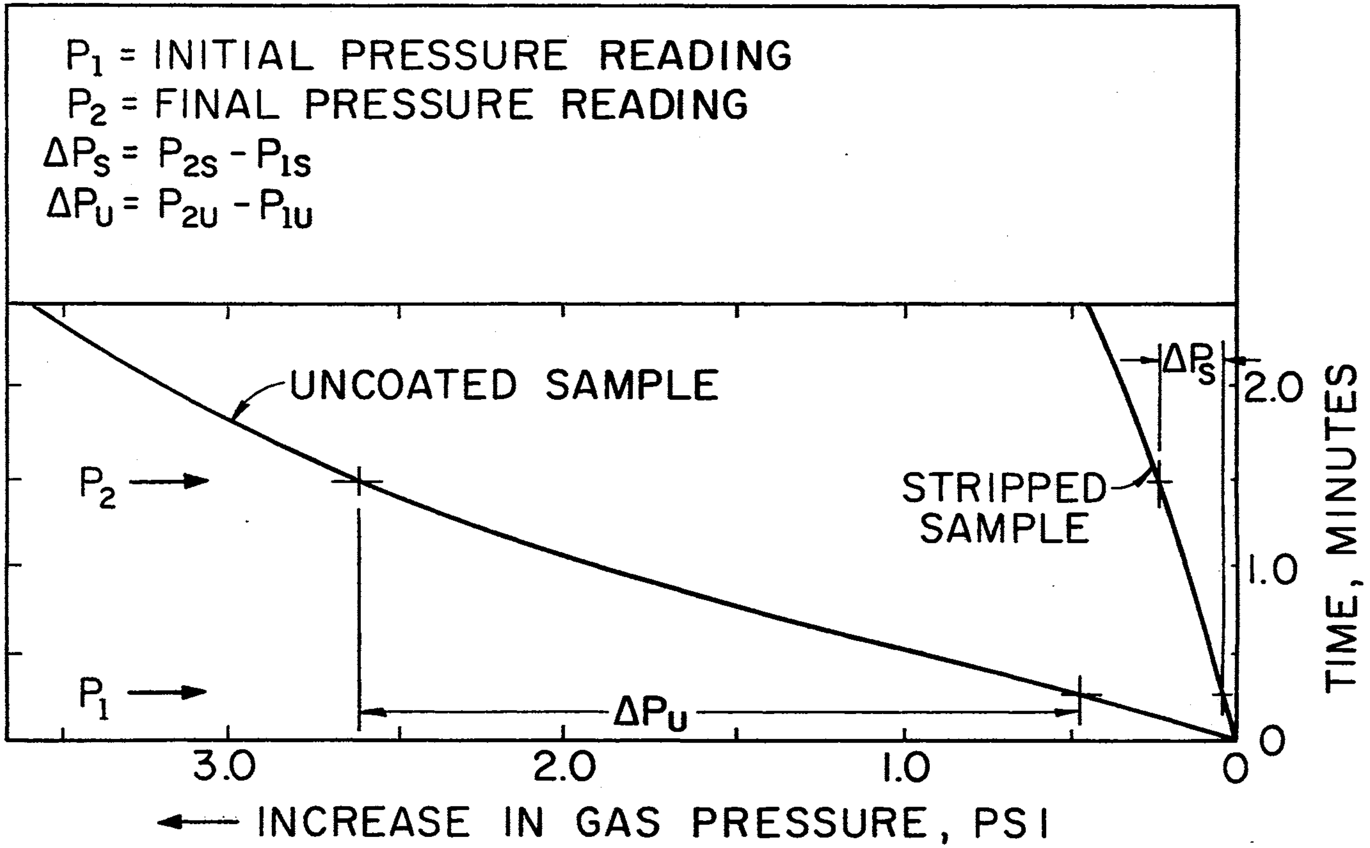


Figure 17. Typical Surface Reaction Test Pressure-Time Curves for Limestone Aggregate, from Strip-Chart Recorder

The change in gas pressure ($\Delta P$) utilized to calculate the amount of asphalt cement coating remaining on the aggregate, and the $\Delta P$ of the uncoated sample are shown in Table XIII for each of the aggregates. These $\Delta P$ values were used to calculate the percentage of asphalt cement coating retained by the aggregate sample, as was illustrated above.

A measure of the precision of the surface reaction test may be obtained using the results of the 4 tests performed on each aggregate sample. The individual $\Delta P$ values, from each SRT, were used to calculate the maximum and minimum retained coatings for each aggregate sample. The differences between these maximum-minimum values were determined and are shown in Table XIII, as the "range" in retained coating for each aggregate. A range in retained coating of less than 1 per cent is noted for 7 of the aggregates, while the Broken Bow sample indicated the largest range at ± 3.6 per cent.

The aggregate retained coatings ranged from 93 per cent for the Stringtown siliceous limestone to 54 per cent for the Broken Bow siliceous gravel. The aggregates classified by type as "limestone", which include the Cooperton, Hartshorne and Stringtown aggregates, had the highest group rating at 89 per cent retained coating. The sandstone aggregates (Cyril, Keota and Onapa) averaged 63 per cent retained coating, while the gravels (Asher, Broken Bow, Gore, and Hugo) averaged 68 per cent retained coating. These results show the same trend for stripping resistance of the aggregates as previously presented and discussed for the immersion-compression test results, where "limestones" were better than "gravels", which were in turn better than "sandstones."

There was low correlation between visually estimated retained coatings of the same stripped aggregate samples as reported for the DIS test (Table XI) and their measured retained coatings from the SRT (Table XIII). The different results obtained by the two methods emphasizes the problem which has long been associated with the visual estimation procedure. Final visual evaluation of the eleven DIS test samples yielded only 5 different retained coatings: 3 at 85 per cent, 3 at 80 per cent, 3 at 75 per cent, 1 at 70 per cent, and 1 at 65 per cent. The SRT results indicated a range of retained coatings from 54 to 93 per cent, with no two aggregates having the same amount of retained coating.

Several factors affect the visual estimation of the amount of retained asphalt coating on the aggregate. These factors include: color of the aggregate, visual perception being limited to the plan or two dimensional view, microscopic breaks in the asphalt coating which are not detectable, cases where the asphalt is in a state of "detachment" and a film of water is between the aggregate surface and the asphalt, and operator error or bias.

The color of the aggregate influences the operators estimation of coating, with lighter colored aggregates being rated lower than darker colored aggregates when both may have about the same amount of asphalt coated surface. This is possible because of the greater contrast between the black asphalt cement coating and the light colored aggregate, which causes the operator to assign a lower value of retained coating to the lighter colored material.

This color factor may be the reason that the Hartshorne limestone and Gore siliceous gravel (both fairly light colored) were visually rated about 5 to 10 per cent lower than the results of the SRT indicated. While, the darker colored Keota and Onapa sandstone were rated about 5 to 10 per cent higher than the SRT results indicated.

The standard method of visual estimation considers only the exposed aggregate surface which appears in plan view. When the stripped specimens were examined from the side and bottom, additional exposed areas were evident. The acid solution used in the SRT reacts with all exposed surfaces, and thus the results are an indication of the total surface area stripped.

By unaided visual examination there appeared to be no disturbance to the aggregate surface where the asphalt coating remained in place. However, under the microscope (30X) numerous pin holes or small breaks in the asphalt coating could be seen. After the SRT, there was evidence of etching or surface reaction at these small discontinuities.

Perhaps the most important factor in the visual estimation method is the operator, i.e., the person performing the evaluation. Repeatability of visual estimation of the aggregate surface area coating was examined by Brown, et al. (11). In this work, four experienced operators were used to evaluate 36 different test samples (DIS test) of partially coated aggregates. Their average range in estimating retained coatings was 16 per cent, and their differences ranged from 2 to 32 per cent. Roediger (37) reported the results of a cooperative stripping test visual estimation project where 10 laboratories estimated the amount of stripping evidenced by 22 different samples. Their visual estimation of precentage retained coating for the same test specimen ranged from 4 to 44 per cent, with an average difference between laboratories of 24 per cent. Different operators were noted to agree more closely when the amount of stripping was small.

The standard ASTM static immersion stripping test results are to be reported only as above or below the 95 per cent retained coating level

(8). Use of this criteria would result in grouping all of the eleven aggregates under study into one group, and classifying them as having less than 95 per cent retained coating. It is obvious that no useful information regarding relative aggregate stripping tendency would be obtained from visual evaluation in accordance with the ASTM method.

All of the factors previously discussed: aggregate color, operator perception, and operator error or bias, were eliminated when stripping was evaluated by the surface reaction test. Further, the results obtained in the SRT are expected to vary less than ± 4 per cent from duplicate tests, and thus are more indicative of the relative stripping tendency of an aggregate than results from the visual estimation method.

The quantities of acids used in each surface reaction test are shown in Table XIV. The normalities of each test solution, except for the HCl and HF mixture, are also given in Table XIV. The concentration of the reagent HCl was assayed at 38 per cent. The assay concentration of the reagent HF varied from 49.1 to 52.0 per cent, as the HF reagent was obtained in 1 lb containers from different suppliers. The relative concentration of acid in each test solution is also indicated by their densities as shown in Table XIV.

## Summary

The results of the immersion-compression, static immersion at 140 F and surface reaction tests for the eleven aggregate samples are summarized in Table XV. Test values for the dynamic immersion stripping test are not included since the surface reaction test values are based on the stripping induced by this test. The 4 per cent I-C retained strengths

## TABLE XIV

## REAGENT DATA FOR SURFACE REACTION TESTS

| Sample | Quantities for 200 ml Test Solution (68F) | | | Normality | Density g/ml (68F) |
|---|---|---|---|---|---|
| | $H_2O$[1] (ml) | HCl (ml) | HF (ml) | | |
| | Limestone Aggregates | | | | |
| Cooperton | 184 | 16 | 0 | 0.974 | 1.014 |
| Hartshorne | 184 | 16 | 0 | 0.974 | 1.014 |
| | Mixed $CaCO_3$ and $SiO_2$ Aggregates | | | | |
| Stringtown | 119 | 54 | 27 | -- | 1.077 |
| Cyril | 119 | 54 | 27 | -- | 1.077 |
| | Siliceous Aggregates | | | | |
| Keota | 35 | 0 | 165 | 22.8 | 1.146 |
| Asher | 35 | 0 | 165 | 22.8 | 1.146 |
| Onapa | 35 | 0 | 165 | 25.2 | 1.159 |
| Broken Bow | 35 | 0 | 165 | 22.7 | 1.142 |
| Miami | 35 | 0 | 165 | 22.7 | 1.142 |
| Gore | 35 | 0 | 165 | 22.6 | 1.141 |
| Hugo | 35 | 0 | 165 | 22.6 | 1.141 |

[1] Distilled water with pH = 5.4.

# TABLE XV

## SUMMARY OF STRIPPING TESTS RESULTS AND AGGREGATE RANKING

| Aggregate | Relative[1] Immersion-Compression 4% AC | Static Immersion 18 hr. @ 140 F | DIS & Surface Reaction | Average | Rank |
|---|---|---|---|---|---|
| | % Ret. Str. | % Ret. Ct. | % Ret. Ct. | % | |
| Cooperton | 100 | 85 | 90 | 91.7 | 1 |
| Hartshorne | 89 | 75 | 85 | 83.0 | 3 |
| Stringtown | 83 | 65 | 93 | 80.3 | 5 |
| Cyril | 77 | 60 | 64 | 67.0 | 8 |
| Keota | 70 | 50 | 56 | 58.7 | 9 |
| Onapa | 58 | 50 | 68 | 58.7 | 10 |
| Asher | 83 | 90 | 74 | 82.3 | 4 |
| Broken Bow | 82 | 90 | 54 | 75.3 | 6 |
| Gore | 70 | 40 | 65 | 58.3 | 11 |
| Hugo | 87 | 95 | 78 | 86.7 | 2 |
| Miami | 78 | 70 | 60 | 69.3 | 7 |

[1] Relative immersion-compression values based on Cooperton limestone having a retained strength of 100 per cent (Table X).

(Table X) were adjusted so that the relative maximum retained strength was 100 per cent, i.e., all of the 4 per cent I-C values were divided by 1.14.

It is realized that these three tests are independent measures of stripping and while their results are not comparable with regard to units the results do provide some insight to the relative stripping tendencies of the aggregates. Based on an average of the three test values, a relative ranking of each aggregate was developed. This is presented in Table XV.

In a consensus, these average test values provide the following aggregate ranking in order of their resistance to stripping: 1) Cooperton limestone, 2) Hugo chert gravel, 3) Hartshorne limestone, 4) Asher chert gravel, 4) Stringtown siliceous limestone, 6) Broken Bow siliceous gravel, 7) Miami chert, 8) Cyril calcareous sandstone, 9) Keota siliceous sandstone, 10) Onapa siliceous sandstone, and 11) Gore siliceous gravel.

A comparison of the stripping tendency from these laboratory test results on Oklahoma aggregates with a standard value is desirable. The best standard value would be from their service record based on field performance. However, no performance data for these various aggregates are available.

Another judgment basis would be a comparison with results in the literature, where the same or similar aggregates have been evaluated. Lang and Thomas (30) compared different generic named aggregates on the basis of visual estimation from a dynamic immersion test. Their results (Table I) show the highest retained coating as being 99 per cent for a "mixed" gravel, while their limestone aggregate showed only a 95 per cent retained coating. Andersland and Goetz (52) found little difference between Lafayette Gravel and Greencastle Limestone after 1 day immersion when tested by the static immersion, immersion-compression and sonic tests. Their Massachusetts Rhyolite (Table III), at the end of 24 hours immersion, indicated better resistance to stripping than either their limestone or gravel as shown by the immersion-compression and sonic test results. Mathews et al. (48) reported the results from a 6 year field observation of 16 different aggregates used for surface treated pavements. The amount of stripped surfacing requiring repairs during this 6 year study showed 80 per cent of the siliceous aggregates (flint gravel, quartzite, and granite) required maintenance, while the basic aggregates (limestone and basalt) required no maintenance.

In general, typical values from the literature indicate that "limestone" should be more resistant to stripping than a "gravel" or siliceous aggregate. Results from this study on Oklahoma aggregates agree, in part, with the literature. In the rating system used, Cooperton limestone ranked highest, however, the Hugo chert gravel was ranked above the Hartshorne limestone and Stringtown siliceous limestone, while the Asher chert gravel was rated almost as high as the Hartshorne limestone. However, the lowest rated aggregates (Keota sandstone, Onapa sandstone and Gore gravel) were highly siliceous.

The average test results of Table XV are illustrated on the bar chart of Figure 18. For the purpose of comparing results of the different stripping tests, the I-C test results may be considered as the "true" relative stripping tendency of the individual aggregates in the study. On this basis, the surface reaction test values (Figure 18)

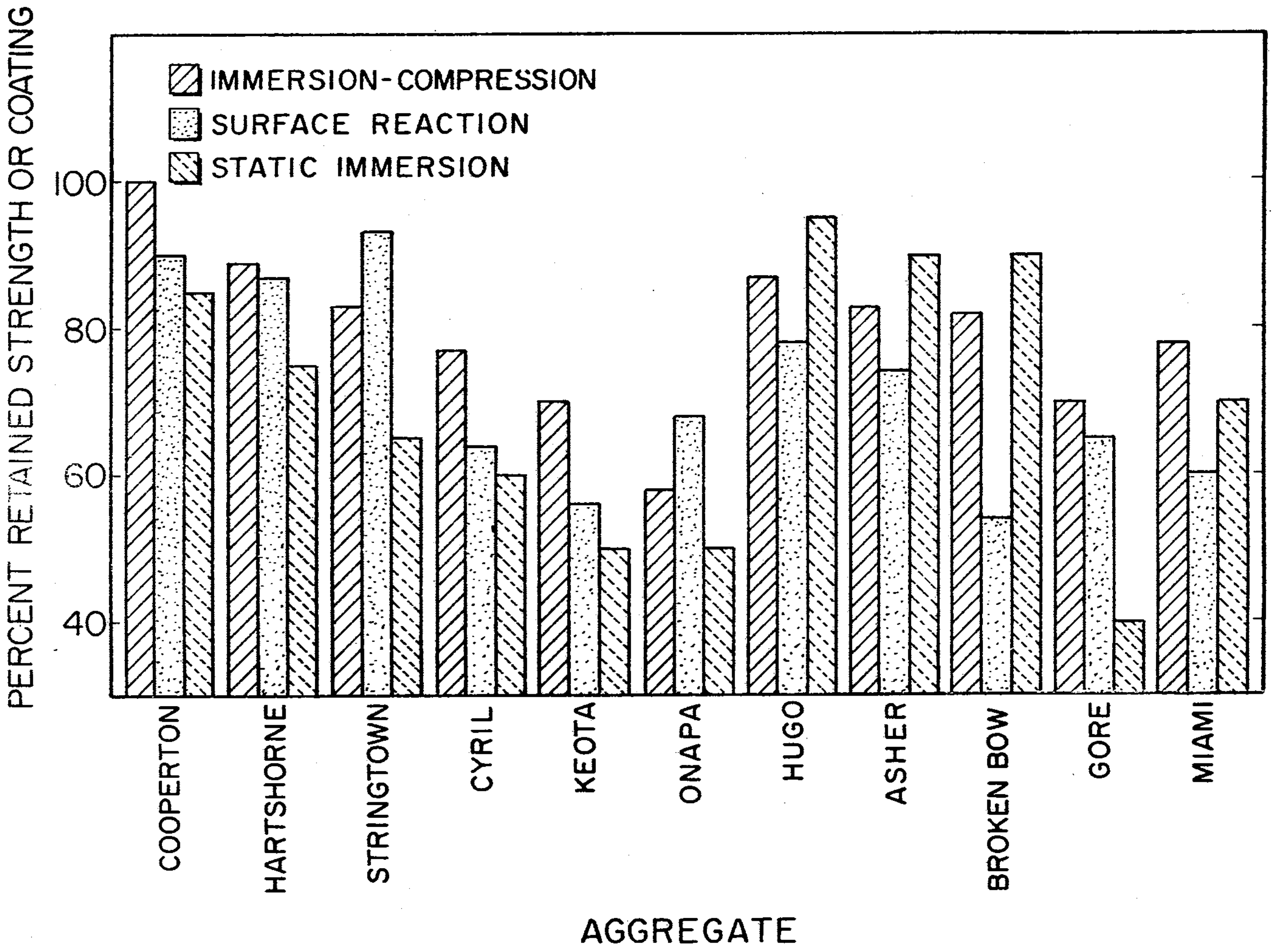


Figure 18. Comparison of Individual Aggregates Stripping Results

agree with the I-C test values for these aggregates: Cooperton limestone, Hartshorne limestone, Stringtown siliceous limestone, Cyril calcareous sandstone, Keota sandstone, and Gore siliceous gravel. Both the surface reaction test and the static immersion test values differ about the same amount from the I-C test values for these aggregates: Onapa sandstone, Hugo chert gravel, and Asher chert gravel. The SIS test values agree with the immersion-compression test results for the Broken Bow siliceous gravel and the Miami chert.

The bar graph of Figure 18 illustrates there is considerable variation of an aggregates stripping tendency as determined by the immersion-compression, static immersion and dynamic immersion (SRT evaluated) tests. However, each test condition was different and perfect correlation was not anticipated. The surface reaction test does provide a fairly simple means of evaluating, in a quantitative manner, the amount of stripping evidenced by asphalt coated aggregate particles which have been subjected to the stripping effects of water.

# CHAPTER VI

# CONCLUSIONS AND RECOMMENDATIONS

## Conclusions

Based on the test procedures employed and the materials used in this study, the following conclusions are made:

1) The surface reaction test provides a quantitative measure of exposed surface area in a stripped aggregate sample.

2) With proper calibration, the surface reaction test can be used to determine the apparent surface area of an aggregate sample in the coarse range of sizes normally employed in paving mixtures.

3) Air void content of compacted bituminous mixtures greatly influence the results of the immersion-compression test. Specimens with less than three per cent air voids had no loss of strength when tested by the modified immersion-compression test method.

4) The modified immersion-compression test method can be used to evaluate mixtures conforming to Oklahoma Highway Department surface course specifications.

5) The static immersion stripping test, using 85-100 penetration grade asphalt cement, will not indicate relative aggregate stripping when the test is performed at 77 F; however, useful results were obtained when the immersion water temperature was held at 140 F for 18 hours.

6) The association of the generic name of an aggregate with its stripping resistance was shown not to be applicable to the aggregates evaluated in this study. However, the limestones (Cooperton, Hartshorne and Stringtown) were more resistant to stripping than the sandstones (Cyril, Keota and Onapa). Also, the gravels (Broken Bow and Gore) containing appreciable amounts of quartz were more susceptible to stripping than the gravels (Asher and Hugo) composed mainly of chert.

## Recommendations

In view of the results of this research work, the following recommendations for further study are made:

1) Perform a field evaluation of these same eleven aggregates and determine their actual performance in an asphalt pavement test section. The validity of the laboratory test results and the relative rankings assigned to the respective aggregates could then be ascertained. Knowledge of how well the aggregates performed under service conditions could be of great assistance in devising laboratory stripping tests that would better correlate with field performance.

2) Further investigation should be given to the method or procedure of inducing stripping on asphalt cement coated particles. Elevation of the water temperature used in the dynamic immersion stripping procedure would result in greater stripping per revolution of the specimen. This would tend to reduce some of the undesirable abrading action undergone by the coated aggregate sample. This type of study could be correlated with asphalt viscosities at different temperatures.

3) Investigate the use of other reagents for the surface reaction test. A more reactive reagent which would create greater gas pressures

when used with siliceous aggregates would be desirable. The use of a smaller pressure vessel, about one-half the size of the container used in this study, would result in higher pressures and greater measurement precision.

4) Investigate the effects of "additives" for increasing the stripping resistance of these eleven Oklahoma aggregates. Results of the immersion-compression test and surface reaction test should be correlated with the effect of additives on stability of the mixtures.

5) Extend the surface reaction test method study to include other types of aggregates, e.g., igneous and metamorphic rocks, and perform additional tests on the eleven Oklahoma aggregates. Obtain sufficient data for a statistical analysis in order to propose adoption of the surface reaction test method as a tentative ASTM standard method for evaluating stripping.

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# APPENDIX

## METHOD OF TEST FOR TOTAL INSOLUBLE RESIDUE IN COARSE AGGREGATE

OHD-L-25 6-29-70

Scope: 1. This method of test is intended for the determination of acid insoluble material in aggregates used in asphaltic concrete.

Apparatus: 2. The apparatus for this test will consist of the following:

(a) Half-gallon jars

(b) Hydrochloric Acid Technical Grade

(c) Evaporating Dishes (Vycor 350 ml)

Procedure: 3. (a) Crush sample so that all material is less than one-half inch.

(b) Split the sample to approximately 200 grams, weigh accurately, and place in clean, labeled half-gallon jar.

(c) Add 400 ml of water and slight excess of concentrated hydrochloric acid (approximately one ml per gram of rock) over amount needed to react with available carbonate. Stir mixture over a period of days until all reaction ceases.

(d) Wash the insolubles free of excess ions by filling jar with tap water, allowing all of the material to settle (about 48 hours) and pour off the clear solution. Procedure is repeated three times.

(e) After the third wash cycle, wash the insolubles into an evaporating dish, dry at 100-105°C and weigh.

Report: 4. Report insoluble residues as retained on the #200 sieve as percent of total sample used.

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