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# Use of waste glass as asphaltic concrete aggregate

Charles Wayne Foster

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#### USE OF WASTE GLASS AS

### ASPHALTIC CONCRETE AGGREGATE

435

CHARLES WAYNE FOSTER, 1946-

BY

A

THESIS

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#### ABSTRACT

The problem of solid waste disposal increases each year, with the annual growth in quantity of solid waste making the present means of disposal even more inadequate. Recycling of solid waste materials is an increasingly popular approach to the disposal problem. The glass component of solid waste is a very promising candidate for recycling, and this investigation studies the possibility of using waste glass as an aggregate for bituminous concrete mixtures. Objectives of this research were to determine whether a glass-asphalt mixture could be designed to meet the Marshall design criteria specified by The Asphalt Institute, to study the amount of degradation occurring in a glass-asphalt mixture, and to find whether a glass-asphalt mixture will be resistant to the action of water.

Material for testing was obtained by crushing waste glass and sieving it into various size fractions which were combined to obtain the gradations desired. Tests were conducted on the crushed glass to determine various physical properties. Marshall tests were run on specimens at different asphalt contents to see if the Marshall design criteria could be met for the gradation used. Glass was extracted from some of the specimens tested, and sieve analyses of the extracted glass were used to determine the amount of degradation occurring in the glass-asphalt mixtures. Statistical analyses were made to evaluate the significance of the Marshall test results. Static stripping tests and immersion-compression tests were used to determine the water resistance of the mixtures.

It is possible to design glass-asphalt mixtures which meet the Marshall design criteria. Extraction data indicates that some degradation does occur during laboratory mixing, compacting, and testing. Mixtures consisting only of glass and asphalt cement show no water resistance; however, the addition of a commercial anti-stripping agent improves water resistance without completely eliminating stripping. Hydrated lime gives excellent water resistance and completely eliminates visible stripping.

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#### I. INTRODUCTION

#### A. Statement of the Problem

U.S. Public Health Service statistics show that the United States presently must cope with 3.5 billion tons of solid waste each year (1). Only expenditures for schools and roads presently exceed the  $\frac{4}{4}$ .5 billion per year spent for refuse collection and disposal services (2), and the costs of these services are expected to increase by at least 20 percent annually for the next few years (3). The increase in expenditures is resulting from an increase in the amount of solid waste generated and increased costs of disposal. According to Richard D. Vaughan, Director of the Bureau of Solid Waste Management of the U.S. Public Health Service, in 1920 an average of 2.7 pounds of waste was collected daily from each person in the United States. Today, this figure has grown to 5.3 pounds per day, and it is estimated that by 1980 the per capita waste collection will be 8.0 pounds per day  $(4)$ .

There are presently several methods of solid waste disposal in use, but many have serious disadvantages. The oldest method of disposal is the open dump. Open dumps pollute the air with strong odors, may pollute ground water, and clutter our countryside with appalling eyesores. Incinerators have the potential for handling the solid wastes; however, incinerators presently in use give off air pollutants and leave up to a 20 percent residue of ash which must still be disposed of. Rail hauls to abandoned mines and strip-mine gullies have been promoted by federal studies, but landowners around proposed dumpsites have protested strongly, the number of sites is limited, and transportation expense will increase with site relocations. Sanitary landfills are

perhaps the most promising means for solid waste disposal. However, required specifications such as minimum cover depths are seldom adhered to, thus resulting in a modified open dump. Even if they were built to specifications, they would be only a temporary solution since conveniently located sites for landfills are becoming more scarce in many areas. The search for new landfill sites in remote areas disturbs conservationists due to destruction of marsh habitats for wildlife, and the longer hauls increase cost and operational problems, especially in inclement weather.  $(3, 4)$ 

An increasingly popular approach to the problem of solid waste disposal is to recycle and reuse the waste materials. If "total recycling" were developed, there would be many advantages. Water and air pollution would be lessened with the discontinued use of open dumps, improperly constructed sanitary landfills, and inefficient incinerators. Aesthetic values would be enhanced considerably by the disappearance of open dumps. Demands on increasingly limited sources of natural resources would be reduced, resulting in the extended use of many minerals and different types of timber. Highly valued land near cities would not have to be set aside for disposal sites.

Setting a monetary value on most of the advantages of salvage and reclamation as a solution to the solid waste disposal problem is virtually impossible; yet, past feasibility studies of salvage and reclamation operations have usually been based strictly on economic considerations. Due to the problems of increasing labor costs and decreasing prices for salvage materials, municipal officials have tended to take a dim view of salvage and reclamation as a sound method

of solid waste disposal. In addition, particularly in large cities, public works officials are justifiably apprehensive of reliance on a disposal method which depends upon fluctuating and sometimes non-existent markets for salvage or refuse by-products. If the markets collapse, the city may find itself with quantities of refuse and no method of disposal except an emergency landfill far from the city (5). Therefore, it is necessary to minimize the costs of collecting, transporting, and processing the waste materials, and a steady market for the salvaged materials must be available. Additional aspects to be considered consist of convincing the public of the necessity for recycling, obtaining necessary legislation, and putting the changes into effect (6).

One constituent of solid waste which is a promising prospect for recycling is glass. About 30 billion bottles and jars are produced annually, and the quantity is expected to increase tremendously with the rapid production growth of the "one-way" bottle  $(1)$ . Glass does not burn, rust, or decay; therefore, it is a prime component of incinerator ash residue, accounting for nearly one-half of the residue by weight (7). Glass may also be troublesome if it is not properly crushed when placed in a sanitary landfill due to unbroken bottles and jars occupying an inordinate amount of space in relation to the actual glass volume itself.

The use of waste glass as an aggregate for bituminous concrete mixtures would reduce some of the expenses which would be considered in determining the economic feasibility of recycling the glass portion of solid waste. A steady market would be available for the salvaged

waste glass through the continuous street maintenance programs of cities. With the waste glass being used in the city from which it was collected, there would be a reduction in the transportation expense of waste disposal. Less would be spent on conventional aggregate and the transporting of conventional aggregate if waste glass were used in this manner. At some future date, this source of aggregate may be even more fully realized. Nationally, there is an abundant supply of conventional aggregates suitable for highway construction; however, there are localized areas, and in some cases regions, in which they are not economically available or are becoming depleted. Some of the existing sources of conventional aggregates are becoming unavailable through zoning restrictions, pollution controls, and appreciating land values  $(8)$ . However, as a first step in investigating the potential use of waste glass as an aggregate, it is necessary to determine the properties of glass-asphalt mixtures.

B. Objectives

The objectives of this investigation were threefold:

- 1. Design a dense-graded glass-asphalt mixture which would meet the Marshall design criteria as specified by The Asphalt Institute.
- 2. Determine the amount of degradation occurring during laboratory mixing, compacting, and testing of the glassasphalt mixtures.
- 3. Determine the water resistance of the glass-asphalt mixtures.

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#### II • REVIEW OF LITERATURE

Since no literature could be found describing the use of glass aggregate in bituminous concrete, it was necessary to use references pertaining to conventional aggregate materials and apply this information to the use of glass as an aggregate.

*A.* Properties of Bituminous Mixtures

Before a bituminous mixture is considered for use in the field, it is necessary that several properties of the design mix be evaluated and found to be satisfactory. These properties are stability, durability, flexibility, skid resistance, and workability.

l. Stability

Stability can best be defined as the ability of a bituminous mixture to resist excessive deformation under imposed loads. This property is developed through the interlocking of aggregate particles, friction between aggregate particles, and cohesion of the binder. Stability gives a bituminous pavement the ability to resist rutting and shoving. It is the property given top priority by most designers; however, stability cannot be designed for at the expense of the other properties, especially durability. Experience has shown that a mixture is rarely found to be unsuitable from purely a stability standpoint. Minimum values for adequate stability vary depending on anticipated loads and traffic conditions. (9)

2. Durability

Durability of a bituminous mixture is defined as its ability to withstand the detrimental effects of traffic, water, ice, air, and

temperature changes (9). There are no direct laboratory methods for measuring all facets of durability; however, other mixture properties such as air voids can be used to indicate potential durability, and direct tests such as immersion-compression can be used to assess a particular property such as water resistance. The overall durability is best measured by time in service. (lO)

Several general rules have been established for the design of bituminous mixtures with respect to durability. First, always use the maximum amount of asphalt in the mixture that is consistent with the stability requirements. Second, do not attempt to design primarily for stabilities which are far in excess of the requirements for given traffic conditions. Third, do not use aggregate combinations which require large amounts of mineral filler as their primary source of strength. (9)

One major requirement with respect to durability is that the paving asphalt must remain plastic to be satisfactory. As an asphalt loses plasticity and becomes brittle due to chemical and physical changes, fine cracks develop which may eventually cause the asphalt pavement to break up. (ll) Several factors contribute to the loss of plasticity, the most important of which are oxidation and volatilization. Oxidation is the reaction of oxygen with asphalt, the rate of which depends upon the character of the asphalt and the temperature. This reaction causes hardening of the asphalt both in the plant and in the field. Volatilization is the evaporation of the lighter constituents, a mixture of hydrocarbons, from the asphalt. This phenomena is greatly accelerated by increased temperatures and thus it frequently occurs in

the mixing process where high temperature is combined with violent agitation. Polymerization, thixotropy, syneresis and separation may also contribute to asphalt hardening. Polymerization is the combining of smaller molecular weight hydrocarbons into larger molecules, while thixotropy is a progressive hardening due to the formation of a gel structure within the asphalt over a period of time. Syneresis is the discharging of a thin oily liquid containing either dispersed or dissolved intermediate and heavier bodies. The asphalt hardens due to the loss of some of the lighter oily constituents. Separation is the removal of either the oily constituents, resins, or asphaltenes from the asphalt due to the selective absorption of some porous aggregates on which asphalt films have been placed. (l2) Hardening of the asphalt, as indicated by a decrease in penetration, will give more resistance to shoving or rutting, but it makes the asphalt much more susceptible to cracking (ll).

The durability of a bituminous pavement is highly dependent upon the ability of the asphalt to adhere to the aggregate in the presence of water (ll). Asphalt has practically no affinity for water, and if a water film exists between asphalt and an aggregate surface, adhesion of the asphalt to that surface is prevented. Most aggregates have an affinity for both asphalt and water; however, if the affinity of an aggregate for water is much greater than it is for asphalt, under certain conditions water may displace the asphalt film with which the aggregate is coated. Several theories have been proposed which explain bond formation between asphalt and aggregate surfaces, with the most commonly followed being that submitted by Winterkorn (13). He interprets the bond formation as a function of the amount and the character of the polar particles present in the asphalt, and he explains the attraction of water by the surface of the aggregate as being primarily due to the electrical charges on the surface of the aggregate and to the dipole nature of' the water molecule. Mertens and Wright  $(14)$  furthered this theory by suggesting that aggregates range from electropositive (predominance of positive charges on the surface) to electronegative (predominance of negative charges on the surface). Thus, if an asphalt is acidic, it will bond best to an electropositive aggregate while a basic asphalt will bond best to an electronegative aggregate.

Two approaches to the problem of' poor asphalt bonding have developed. The first method is to chemically treat the surfaces of the aggregates with thin coatings of certain substances which are insoluble in water but soluble in asphalt. The second method involves treating the asphalt with oil soluble or readily miscible compounds to increase the quantity of dipoles in the asphalt. In addition, Dow  $(15)$ has found that the addition of hydrated lime is very effective in preventing the stripping of asphalt from some aggregates.

3. Flexibility

Flexibility is concerned with the ability of a bituminous pavement to deform somewhat without cracking. The adjustment capacity of a pavement refers to how well the pavement will follow settlements of its underlying base. This is of great interest when the pavement is constructed on a flexible or compactible base rather than a rigid base. Fatigue resistance refers to the ability of the pavement to

bend repeatedly without fracture. For dense-graded mixtures investigated by Monismith  $(16)$ , the fatigue effect decreases with increasing asphalt content and it becomes more pronounced with increasing magnitudes of load. (l7)

# 4. Skid Resistance

Rader  $(17)$  defines skid resistance as the ability of a pavement surface to provide friction for the deceleration of a sliding object. Sufficient voids should be provided in the compressed bituminous pavement to allow for increased densification by traffic loads and for expansion of the asphalt caused by temperature increase so that exuding of asphalt on the surface of the pavement will be avoided.

5. Workability

The final property to be considered is workability of a bituminous mixture during construction operations. The degree of workability required depends upon the conditions of use and the type of equipment available. For example, highway pavements with little handwork do not need to be as workable as municipal pavements that require much more hand placing and raking. For easy placement in uniform layers with sufficient densification, bituminous paving mixtures must be workable at the temperature desired. Ease in handling cannot be at the expense of density and stability of the mix.  $(17)$ 

B. Factors Affecting the Properties of Bituminous Mixtures

The properties of a bituminous mixture are affected by many factors including the following: type, quality, surface texture, size, shape, and gradation of the aggregate; quality, consistency, source, and amount of asphalt; amount and type of compaction; and quality and type

of mineral filler. A thorough understanding of the effects of each of the factors mentioned is required in order that results from tests conducted on bituminous mixtures may be accurately evaluated.

#### 1. Stability

The two primary aggregate properties contributing to stabilityaggregate interlock and internal friction-are both improved through the use of aggregates having a rough surface texture; however, Li and Kett (18) have found that the rough surface texture does not exert a significant influence if the bituminous mixture is at or above optimum asphalt content and the aggregates possess a stronger affinity for asphalt than for water. This can result from coating the aggregate particles with asphalt films thick enough to smooth out the roughness, with the stronger affinity of the aggregate particles for asphalt than for water insuring that the coating of asphalt will remain on the particles.

When an excess quantity of asphalt is present, the aggregate framework is destroyed as the individual particles are forced apart. This results in the pavement being unable to carry any appreciable loads without shoving or rutting occurring. However, the asphalt must be present in such quantity as to serve its role as a cohesive binder; otherwise, the aggregate will not remain in position to handle the traffic loads.

High temperatures have an adverse effect on stability due to a resultant decrease in the viscosity of the asphalt which hinders the role of the asphalt as a binder.

Csanyi (19) refers to studies and tests which verify the fact that increasing the quantity of mineral filler tends to increase the stability of a bituminous mixture by improving the internal friction of the mixture through increased particle-to-particle contact. There is a limit to the beneficial aspects of adding mineral filler beyond which durability suffers. The studies also give indications that the type of mineral filler has an effect on stability.

It has been generally accepted for many years that angular aggregate substantially increases the stability of mixtures through increased mechanical interlock of the angular particles. Goetz and Herrin (20) concluded that higher percentages of angular aggregate appreciably increase the stability of open-graded mixtures while having a negligible effect on dense-graded mixes and that angular fine aggregate increases the stability of a mixture more than angular coarse aggregate. The second conclusion by Goetz and Herrin is further supported by Proudley and Waller (21). They concluded that a high quality heavy duty pavement can be constructed with rounded gravel provided that the fine aggregate is angular.

Li and Kett  $(18)$  stated that if a bituminous mixture contains a sufficient proportion of particles whose width to thickness, or length to width, equals or exceeds three to one, its stability is adversely affected. It was concluded that the percentage of flatshaped particles that may be included without causing undesirable effects upon a bituminous mixture is as high as 30 percent and may possibly be 40 percent.

It would appear that the stability values for bituminous mixtures containing glass aggregate would be somewhat lower than those obtained with the use of conventional aggregates. The angularity of the glass particles is favorable; however, the non-porous, highly smooth surface texture and the large quantity of flat and elongated particles would be stability reducing factors.

2. Durability

In order for a coarse aggregate to perform satisfactorily in a pavement, it must be tough enough to withstand the action of rolling during construction and then the action of traffic without breaking under the imposed loads. This property is evaluated with the Los Angeles abrasion test and, usually, the coarse aggregate should have a Los Angeles abrasion loss of not more than 45.

The soundness of an aggregate is a measure of how much the aggregate disintegrates under the action of weather. This property, which is related to the pore structure of the aggregate, is tested by alternately soaking the aggregate in a saturated solution of magnesium sulfate and drying it in an oven. To be satisfactory, an aggregate must not have more than 18 percent loss in five cycles.

The cleanliness and purity of an aggregate is critical to how well asphalt will adhere to the surface of the aggregate particles. Raveling is enhanced by dirty aggregates, particularly those with clay coatings. (11)

All aggregates should be checked to determine whether or not they have a greater affinity for water than for asphalt. This check is conducted through the use of the static stripping test. Those

aggregates showing a greater affinity for water are highly undesirable as they easily lose their coating of asphalt in the presence of water. However, commercial additives are available which retard the stripping action.

The quantity of asphalt present in a bituminous mixture is highly relevant to its service life. An excess of asphalt results in bleeding (exuding of asphalt from the mixture) which leads to failure of the pavement. On the other hand, if asphalt is present only in sufficient quantity to serve its function as a binder then there is a high volume of air voids. In this case, the pavement may still have good resistance to movement, but other factors must be considered. A high volume of voids is conducive to hardening of the asphalt through oxidation which may shorten the life of the pavement. Also, a low asphalt content can result in a brittle pavement which will ravel under the action of traffic. At least two percent of the total volume of a bituminous mixture must be air voids to insure against bleeding, and no more than six percent should be air voids in order to avoid undue hardening of the asphalt and raveling. (ll)

Asphalts of low quality and poor consistency are frequent sources of pavement problems. Use of a very hard asphalt produces a brittle pavement which results in raveling and excess cracking. Another aspect of asphalts which is frequently overlooked is the variance of asphalt properties due to different sources of material. Consideration of these factors assists in prolonging the service life of a pavement.

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An increase in compactive energy reduces the amount of asphalt required to minimize the voids due to a reduction of void spaces between the aggregate particles. Too much compaction will decrease the voids below the acceptable minimum and could result in the bleeding of asphalt which was considered to have been of a sufficient amount. It is imperative to realize that an asphalt pavement ultimately becomes further consolidated under traffic loads so that the compactive effort will be proportioned to allow for this additional compaction.  $(22)$  If too little compaction is given to the bituminous mixture, an excess of voids will result which is also detrimental to the pavement, as explained previously.

Csanyi (19) found that as the mineral filler content increases, the brittleness and tendency of the mix to dry out and crack in service also increases. Experience has indicated that not only the quantity of mineral filler present but also the manner in which the particles are coated has some bearing on the durability of the *mix*  in service. When the filler particles are individually coated with thin films of asphalt, indications are that good durability can be obtained. When agglomerations of filler particles are coated, the agglomerations break down under traffic, releasing the individual filler particles of the agglomeration to adsorb asphalt from the mix and thereby cause a drying of the *mix* with an attendant decrease in durability.

Pauls and Goode (23) show that the quality and quantity of sands and mineral fillers have considerable effect on the resistance of mixtures to water. A high percentage of fines of good quality may often

be used to overcome the adverse effect of a less satisfactory coarse aggregate.

Norman McLeod (24) stated that a dense-graded pavement will often lack the desired voids, but by changing the gradation, voids can be increased so that the resulting pavement is more durable.

The durability of bituminous mixtures containing glass aggregate should be favorably affected by the non-porous structure of the glass which minimizes problems of soundness and separation. Due to the nonporous structure, lower temperatures should be required in the drier to drive off moisture, and thus lower mixing temperatures should also be possible. This reduces the chances for volatilization of the asphalt. The smooth surface texture of glass, however, hinders good bonding with asphalt, and although glass has an electronegative charge, asphalt is weakly polarized and no strong chemical attraction is available for bonding. The lower asphalt contents which may be required to obtain sufficient stability with glass aggregate may reduce the asphalt film on aggregate particles and thereby lead to a reduction in durability.

3. Flexibility

Two of the primary factors influencing the flexibility characteristics of a bituminous mixture are the physical and chemical properties of the asphalt. One of the most important of these properties is the viscosity of the asphalt, with a less viscous asphalt generally producing a more flexible bituminous mixture. The change in viscosity with time must not be so great that the asphalt hardens to the point of being brittle. Also, the temperature susceptibility of

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the asphalt should be low enough that few opportunities for cracking are available. Ideally, from the standpoint of flexure, an asphalt should have practically no change in viscosity over the range of temperatures encountered in the pavement structure and should be of sufficient consistency to bind the aggregate particles together to prevent raveling. An asphalt must have sufficient adhesive ability to bind to the aggregate particles so that failure under flexure will not occur at the asphalt-aggregate interface. Also, since the asphalt is subjected to tensile stresses, it must be ductile in order that the material can elongate without fracture at the stress levels imposed.

Generally, the more open the gradation the more flexible is the mixture. The asphalt in these mixtures, although used in smaller quantities, would exist in thicker films, thereby giving increased resistance to fatigue cracking. Also, the aggregate must be resistant to degradation and fracture which would contribute to reduced flexural strength due to planes of weakness being developed, and it must be able to develop good adhesion with the asphalt.

A final factor to be considered is that the cumulative effect of an increase in the density of a pavement along with increased resiliency of its subgrade due to an increase in water content will yield a strong possibility of cracking of the pavement. (l6)

Flexibility of bituminous mixtures using glass aggregate may be low. It would seem that the flat and elongated particles would be easier to degrade and fracture under imposed loads. The lower asphalt contents which may be required for adequate stabilities might also endanger flexibility due to a decrease in material which can

deflect with imposed loads. Weak bonding between the glass and the asphalt can lead to problems if the loads are concentrated enough to cause a failure at the glass-asphalt interface. Poor resistance against the action of water is troublesome because when the asphalt is stripped from the aggregate, the mixture loses flexibility.

4. Skid Resistance

All solid particles offer resistance to sliding objects; however, the amount of resistance developed by an aggregate depends upon the surface texture of the aggregate particles and the amount of pressure exerted upon the sliding object. Higher resistances are developed by aggregates with rough surface textures.

An excess of asphalt must be avoided by providing enough air voids to allow for expansion of the asphalt due to high temperatures and by allowing for further densification of a pavement by traffic. Care must also be taken that no excess asphalt remains on the surface of a pavement immediately after construction.

Moyer (25) found that the friction values for rounded aggregate were about 25 percent lower than for angular aggregate in the wet tests.

The use of glass aggregate may affect the skid resistance of bituminous mixtures in that the smooth surface texture of glass will decrease friction while its angularity will increase friction. The net effect can probably be determined only through field testing.

5. Workability

A large percentage of coarse particles will produce a harsh mix that complicates construction operations, whereas, fine-grained mixes are more workable but lack the stability that is developed in a wellgraded aggregate combination.

Bituminous mixes containing aggregates with rough surface textures are difficult to place due to frictional resistance. For this same reason, angular aggregates will also decrease the workability of a bituminous mixture. (26) Also, mixtures with excess asphalt and/or mineral filler are difficult to place.

The workability of bituminous mixtures containing glass aggregate may be improved by the smooth surface texture of glass which will assist in obtaining adequate compaction. On the other hand, the angularity, flatness, and elongation of glass particles may produce harsh mixtures which will not compact well. Thinner films of asphalt on the glass particles due to lower asphalt contents which may be necessary for adequate stability may also result in more difficult compaction due to an increase of interparticle friction.

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#### III. EXPERIMENTAL PROCEDURES

#### A. Materials

All of the glass aggregates used for this study were obtained by crushing waste glass consisting primarily of ''one-way" beer and soft drink bottles. The initial treatment of the bottles consisted of a hot bath where labels and all other foreign materials were removed. After allowing the bottles to dry, they were fed through a jaw crusher for initial breaking and then passed through a roller mill to obtain finer sizes. The crushed glass was separated into nine different size fractions by sieving; the sizes ranged from material passing the  $1/2$ -in. sieve and retained on the  $3/8$ -in. sieve to material passing the No. 200 sieve. A washed sieve analysis was run on random samples from each of the size fractions, and the results were used to determine the quantity required of each fraction to obtain the desired gradation.

Random samples from the larger size fractions were tested for percentage of flat and/or elongated particles using Corps of Engineers Methods CRO-C ll9-53 and CRO-C l20-55. One hundred to three hundred particles of each of the larger size fractions were obtained by subdivision of the random samples with the larger particle sample size being used for the smaller size fractions. The length, width, and thickness of the particles were measured, and the particles were classified based on the ratios of length to width and width to thickness. A flat particle was defined as having a width to thickness ratio of three or greater, and an elongated particle was defined as having a length to width ratio of three or greater. Results showed

that nearly all the particles in the  $1/2$ -in. to  $3/8$ -in. size fraction were flat, but the percentage of flat and elongated particles decreased as the size of the sieve openings on which the particles were retained approached the wall thickness of the bottles. The percentage of flat particles began to increase again with the material passing the No. 16 sieve, and a microscopic investigation of the material passing the No. 30 sieve indicated the presence of a significant amount of flat and elongated particles.

The hardness of glass aggregate was tested using the Los Angeles abrasion test (AASHO T-96). HYdrometer analyses were run on the glass passing the  $N$ . 200 sieve to determine the properties of that fraction, and the bulk specific gravity of glass was also determined. Information obtained from these tests is shown in Table I.

The asphalt used in this research was an  $85-100$  penetration asphalt cement donated by the Shell Oil Company. It was produced from a West Texas crude oil. Properties of the asphalt cement are listed in Table II.

Three commercial anti-stripping additives and hydrated lime were used for the water resistance studies. All three additives may be chemically categorized as proprietary cationic and oil soluble surface active agents. The composition of additive A is not available; however, additive B is described as an amidoamine soap, and additive C is an ester of crude tall oil and triethanolamine. The hydrated lime used conforms to ASTM C206 (Type S) and ASTM C207 (Types S and N).

#### B. Marshall Test Procedures

The Marshall test was conducted according to procedures specified by ASTM D 1559 with the following exceptions:

- l. Immediately after mixing for two minutes in the Hobart Model N-50 mixer, the bituminous mixture was placed in compaction molds. The molds were retained for 30 minutes in an oven kept at 275F to insure a uniform temperature for all specimens at compaction. At the end of this 30 minute retention period, the molds were removed and the mixtures were spaded and compacted according to specifications.
- 2. In the first trial mix series, the specimens were placed in plastic bags during immersion in the l40F water bath for 30 minutes in an attempt to prevent stripping which would affect the results of the extraction analysis of the aggregate. Most specimens were affected by leakage of the plastic bags; therefore, the second trial mix series was placed in an oven maintained at a constant temperature of l40F for two hours before being subjected to the Marshall test rather than being immersed for 30 minutes.

#### C. Degradation Tests

The asphalt was removed from the specimens with a reflux extractor using a benzene solvent. A washed sieve analysis was then run on the retained aggregate according to procedures specified by AASHO T30. The results were compared with the original quantities of the size fractions to determine the amount of degradation occurring during laboratory mixing, compacting, and testing.

### D. Water Resistance Tests

Initial studies of the ability of a glass-asphalt mixture to resist the action of water were conducted using static stripping tests according to the procedures outlined in ASTM D 1664. Asphalt and glass were heated separately and then mixed until the glass was thoroughly coated. The mixture was oven-cured, remixed until no bare spots were visible, and allowed to cool to room temperature. The mixture was then placed in distilled water at room temperature for 16 to 18 hours. Visual examination of the mixture was used to estimate the percentage of film retention. At least 95 percent of the film must be retained for the mix to be satisfactory.

Further testing consisted of studying the effects of water on compacted specimens of glass-asphalt mixtures. These tests were run in accordance with the provisions of ASTM D 1075. Six 4 by 4-in. cylindrical specimens were made for each test according to the procedures of ASTM D 1074 with the exception that no 'butter batch" was used due to the limited amount of glass available. After oven-curing, the bulk specific gravity of each of the six specimens was determined, and the six specimens were divided into two groups such that the average bulk specific gravity of group l was essentially the same as that of group 2. Group 1 was then stored in an oven at  $77F$  for four hours as preparation for testing. Group 2 was prepared for testing by immersion for 24 hours at  $140F$  followed by immersion for two hours at 77F. The specimens were each tested in axial compression without lateral support at a uniform rate of vertical deformation of  $0.2$  inch per minute. A linear variable differential transformer was used to

insure a uniform rate of deformation. The effect of water on cohesion is indicated by the loss in strength after the immersion period. For a mixture to have satisfactory water resistance, the average compressive strength of the immersed specimens must be at least 70 percent of the average compressive strength of the specimens prepared in the air bath.

#### IV. TEST RESULTS

#### A. Mix Design

The initial gradation was based on the suggested gradation for maximum density presented by Goode and Lufsey (27) and is calculated from the relation

$$
P = (d/D)^{0.45}
$$
 x 100

where,

 $P =$  Percent passing a sieve having an opening of d inches,

D = Maximum size of the aggregate, and

0.45 = An empirical constant.

The 0.45 exponent for maximum density was based upon reasonably equidimensional particles; therefore, it was realized that this exponent might not provide maximum density for the flat and elongated glass particles. However, it was used as a starting point and modified as needed. The initial gradation used is shown in Table III as Gradation No. l.

Six asphalt contents were selected in 0.5 percent increments from  $4.5$  to 7.0 percent (total weight basis), and five specimens were made for each asphalt content. Six specimens were made daily for five days with one specimen representing each of the six asphalt contents being made each day. The Marshall specimens were compacted on each end with 50 blows by a lO-pound weight dropped l8 inches. The bulk specific gravity of each specimen was determined according to ASTM D 2726 after which the specimens were immersed in a l40F water bath for

30 minutes and then tested for stability and flow. The percent air voids and the percent voids in the mineral aggregate of each specimen were determined from the bulk specific gravity of each specimen.

The Marshall test results of the specimens made from the initial gradation are given in Table IV and plotted in Figure 1. As can be seen in Figure 1, there was no asphalt content at which all the data would satisfy The Asphalt Institute's suggested Marshall design criteria shown in Table V. Stability and flow were adequate up to the highest asphalt contents; however, there was no asphalt content at which the requirements of both percent air voids and percent voids in the mineral aggregate could be met. Therefore, it became necessary to modify the maximum density gradation.

The second series of tests was conducted on specimens made from Gradation No. 2 which is shown in Table I. It should be noted that this gradation differed from the maximum density gradation in that there was a decrease in the amount of material passing the No. lOO and the No. 200 sieves. For the second series, the lower limit of the asphalt contents was dropped to  $4.0$  percent in an attempt to get a better distribution of data about the projected optimum asphalt content. The same procedures were followed for the second series of tests as those used for the first series of tests, except as noted previously.

The Marshall test results of the specimens made from Gradation No. 2 are given in Table VI and plotted in Figure 2. All Marshall design criteria was satisfied with an asphalt content of 5.5 percent on a total weight basis.

The standard Marshall design method does not include a statistical evaluation of the data to determine whether the differences in mixture properties for different asphalt contents are statistically significant. However, in view of the variation in measured properties for a single asphalt content encountered in early testing, it was decided to design the experiments so that a statistical analysis could be performed. A randomized complete block design was used, with blocking being on the basis of the day on which specimens were made, and the treatments being the varying asphalt contents. An analysis of variance technique was used to determine whether differences in stability, flow, air voids and voids in the mineral aggregate (VMA) were statistically significant at a .05 significance level. Where significant differences were obtained, a Duncan Multiple Range test was used to compare individual values. Results of' these analyses are shown in Table X. Details of the statistical techniques are given in Appendix C.

# B. Degradation Tests

One specimen was chosen at random from the five specimens representing each asphalt content for both test series. The asphalt was extracted, and washed sieve analyses (AASHO T30) were run on the recovered aggregate. Results from this study are presented in Table VII.

The development of the Hudson  $\bar{A}$  factor provided an index of the relative coarseness or fineness of an aggregate which is sensitive enough to detect minor changes in gradation  $(28)$ . Hudson  $\overline{A}$  is a fundamental constant which measures and assesses the effects of

gradation changes by a single number rather than a multiplicity of :percentages. It is simply one-hundredth of the sum of the percentages passing the ten U.S. Standard sieves from the 1  $1/2$ -in. through the No. 200 sieve. Thus, increases in fineness resulting from degradation are reflected in larger Hudson A values.

The Hudson A values for the first series of tests indicate that there was only a small amount of degradation. The higher asphalt content specimens had less degradation, and most of the degradation affected the material larger than the No.  $8$  sieve. The decrease in the percentage of material passing the No. 200 sieve is considered to be due to stripping and the subsequent loss of material from the specimens during the immersion period prior to the Marshall test for stability and flow.

Degradation is more evident in the test results of the second series of specimens. Here the Hudson  $\bar{A}$  has increased by as much as 0.20, indicating that many new surfaces exist in the bituminous mixtures. The amount of material passing the No. 200 sieve increased by as much as 59 percent. Again, degradation can be seen to be greatest for the larger particles and at the lower asphalt contents. A comparison of the air voids for the two series of tests indicates that the second series has greater air voids.

# C. Water Resistance Studies

Static stripping tests were conducted on several combinations of glass aggregate and bituminous materials. Stripping was quite noticeable when glass aggregates were coated with asphalt cement or coal tar and then subjected to water immersion; however, mixtures of
glass and either a slow-setting cationic emulsion or an asphalt cement with a proprietary anti-stripping compound exhibited no stripping.

*As* a preliminary to the immersion-compression test series, one set of six specimens utilizing Gradation No. 2 and an asphalt content of' 5.5 percent (total weight basis) was made to determine the percentage of stability retained after immersion in  $140F$  water for  $24$  hours.

However, all six specimens completely deteriorated (Figure 4) making stability tests impossible. This re-emphasized the stripping problem and made the study of' water resistance most important to future research into possible use of glass as an aggregate.

Three anti-stripping compounds were initially investigated using the immersion-compression test. Each compound was tested at three different concentration levels of 1, 2, and 4 percent by weight of the asphalt. Six specimens were made for each concentration level of each anti-stripping agent with three of these being tested dry and the other three tested wet. Table VIII contains the compressive test results of the specimens, Figure 3 is a graph of the average strength of the four percent concentration of each additive for both test conditions, and Figure 5 shows the effect of differing levels of antistripping additives on the retained strength of the specimens. It is readily apparent that only the four percent concentration of additive A met the minimum requirement for satisfactory water resistance with the four percent concentration of additive B just failing. However, as can be seen in Figure  $4$ , the specimens containing four percent of additive A still had considerable loss of material due to stripping,

so it was decided to search for an additive which would not only provide adequate strengths after immersion but also keep the specimens intact.

It was reported that hydrated lime reduced the stripping tendency of certain aggregates (29); therefore, a set of six specimens was tested using hydrated lime as the material passing the No. 200 sieve with a four percent concentration of additive A. The results showed the immersed specimens now having higher compressive strengths than the dry specimens and there was absolutely no visible stripping, as can be seen in Figure 4. These encouraging results led to a series of tests devoted to finding the least amount of hydrated lime required to maintain satisfactory retained compressive strengths and to keep specimens intact while immersed in the hot water bath. In an effort to maintain satisfactory air voids, the weight of hydrated lime was decreased in order to obtain the same volume as resulted from the amount of glass normally used as the mineral filler. The weight was decreased due to hydrated lime having a lower specific gravity than that of glass. The test results are given in Table VIII and shown graphically in Figure 3. When one percent of the aggregate by volume was hydrated lime, the compressive strengths of the immersed specimens were essentially the same as those of the specimens from an air bath and the immersed specimens had a negligible amount of material loss due to stripping.

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#### V. DISCUSSION

The data from this research indicates that a bituminous mixture satisfying the Marshall design criteria suggested by The Asphalt Institute can be designed using crushed glass aggregate and a modified maximum density gradation. The stability values obtained by the Marshall testing method were somewhat low; however, the fact that a large percentage of the glass particles were flat and/or elongated suggests that the stabilities might have been on the conservative side due to particle orientation effects. Puzinauskas (30) observed that elongated or flat particles tend to become axially aligned perpendicular to the direction of an applied compactive force. This effect was most pronounced in specimens compacted by intermittent impact-type compactive forces such as are applied by the Marshall compactor. Puzinauskas also found that specimens tested such that the compressive force was applied in a direction parallel to the direction of the applied compactive force always developed higher strengths than specimens tested with the compressive force applied in a direction perpendicular to the direction of the applied compactive force. This would support the suggestion that the Marshall stability values were conservative since the Marshall specimens were tested in a direction perpendicular to the direction of compaction while loads in the field are applied in a direction parallel to the direction of compaction.

Based on the work of Puzinauskas, it would seem that the specimens tested in direct compression (compressive force applied parallel to compactive force) would be more likely to develop strengths in excess

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of required values. The laboratory data for the immersion-compression specimens does not verify this, however. Strengths of both dry and wet specimens were below the suggested minimum requirements for direct compression testing. The minimum requirements are shown in Table IX. This can be partially explained by the results of an extraction test (Table XI) which was conducted on an immersion-compression specimen and shows more degradation of the glass by compacting with a constant pressure of 3000 psi for two minutes than is obtained by the impacttype of compaction by the Marshall hand compactor. Since, according to Puzinauskas, compaction by a constant pressure creates less particle orientation than the impact type of compaction, the immersion-compression specimens probably did not exhibit orientation effects to the degree found in Marshall test specimens. It is also possible that the strength requirements for mixtures designed by the direct compression method are more stringent than those for mixtures designed using the Marshall method. Since the two methods for preparing and testing specimens are so different, a direct comparison of results is difficult.

The statistical analysis of the Marshall test data (Table X) indicates that there are some significant differences among the mean values for each asphalt content. This was true regardless of whether stability, flow, air voids, or VMA values were compared. However, all means for a property such as stability were not significantly different. As an example, in Series 2, the stability at 5.5 percent asphalt is not significantly different from the stability at 5.0 percent. In the same series, the flow value at 5.5 percent is not

significantly different from the flow value at 5.0 percent. This would seem to indicate that, for the particular glass gradation investigated, changes in asphalt content of up to one-half percent may not result in significant changes in at least some mixture properties. Thus, the asphalt content of a mixture can vary somewhat from optimum without appreciably altering some of the mixture properties.

The data also indicates that degradation of the aggregate may be a problem in mixes deviating from a maximum density gradation. Nijboer (3l) reports that a German investigator, Herrmann, found the crushing of aggregate under traffic to be dependent upon the grading, with the maximum density gradation resulting in less crushing. Laboratory tests for this research support the report of Herrmann. Degradation of the modified maximum density gradation specimens was greater than that of the maximum density gradation specimens as was seen by an increased Hudson A and an increase in material passing the No. 200 sieve. The modified maximum density gradation specimens had an increase in percent air voids which could have resulted in less cushioning of the aggregate from the impact of the compaction hammer which caused greater degradation of the aggregate particles.

How much the increased degradation of Gradation No. 2 would harm field performance is not known. It is possible that field compaction by rolling will not result in as much degradation as the impact type of compaction used in the laboratory; however, the final effect of degradation will have to be determined after analyzing field installations of the glass-asphalt mixture.

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From the results of the immersion-compression tests, it is expected that stripping can be prevented by the addition of hydrated lime to the glass aggregate. One percent hydrated lime by volume of the glass aggregate was found to be an adequate amount to prevent stripping. Effective mixtures should also be available when four percent of the asphalt weight consists of additive A with no hydrated lime added. These quantities are based upon the use of the asphalt cement employed in this investigation. Since asphalt cements from different crude oils may exhibit different chemical compositions, it may be necessary to determine optimum amounts of hydrated lime or anti-stripping agents on a job-to-job basis when different asphalt cements are used.

Increased strengths of the immersed specimens containing hydrated lime might possibly be explained by the  $24$  hour immersion of the hydrated lime specimens in the l40F water bath. A pozzolanic reaction between the hydrated lime and finely crushed glass in the presence of water would result in the formation of a cementitious silicate gel. While most pozzolanic reactions proceed rather slowly, the 140F temperature would have an accelerating effect.

Some comments are in order regarding the economics of using hydrated lime or a commercial anti-stripping agent to improve water resistance. A recent issue of Engineering News-Record (32) gives prices for hydrated lime in major cities throughout the United States. The average cost of commercial hydrated lime was  $$39.53$  per ton, F.O.B. plant. Prices ranged from \$21.70 per ton to \$65.50 per ton, F.O.B. plant. The average cost of the three commercial additives was \$300.00

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per ton, F.O.B.. Use of a commercial additive comprising four percent by weight of the asphalt would increase the cost of a one ton batch of the glass-asphalt mixture by \$0.66, assuming an asphalt content of 5.5 percent. Use of commercial hydrated lime comprising one percent by volume of the glass aggregate would increase the cost of the glassasphalt mixture by \$0.38 (using \$39-53 per ton as the cost of hydrated lime). Since the use of hydrated lime completely eliminated visible stripping while the use of the anti-stripping additive permitted some stripping to occur, hydrated lime is the preferred additive. However, one other factor to be considered is the ease with which the additive can be introduced into the mixture. Commercial anti-stripping compounds can be blended with the asphalt cement by the asphalt supplier while the use of hydrated lime will require a mineral filler feeder device at the plant.

 $3<sup>4</sup>$ 

#### VI. CONCLUSIONS AND RECOMMENDED FURTHER RESEARCH

Based upon the laboratory work carried out in this study, the following conclusions have been reached:

- l. Bituminous mixtures satisfying Marshall design criteria recommended by The Asphalt Institute can be designed using penetration grade asphalts and aggregates composed entirely of crushed glass.
- 2. Some degradation of the glass aggregate occurs under laboratory mixing, compacting, and testing conditions. The degradation appears to increase as the gradation deviates from a maximum density curve.
- 3. Severe stripping occurs when a bituminous mixture using densegraded glass aggregates and asphalt cement with no additives is exposed to l40F water for 24 hours.
- 4. Mixtures of glass aggregate and asphalt cement treated with an anti-stripping compound will not strip when subjected to the static stripping test. Mixtures of glass aggregate and a slow-setting cationic emulsion are also resistant to stripping in the static stripping test.
- 5. Mixtures of glass aggregate and asphalt cement treated with an anti-stripping compound will meet requirements of the immersion-compression test (ASTM D l075) provided that adequate amounts of the compound are used. However, some stripping does occur even when the compounds are used.
- 6. Mixtures of glass aggregate with hydrated lime mineral filler and asphalt cement will meet minimum requirements for percent

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retained strength in the immersion-compression test and show no visual evidence of stripping.

The fact that strengths of glass-asphalt mixtures tested in direct compression were lower than required values for this property indicates that mix design using this test method may require further modification of the grading and asphalt content. The indication that degradation is more severe under static compression forces also points up the need for an investigation of degradation under field compaction procedures. A field installation would permit assessment of degradation and would also provide information relative to ease of placement, skid resistance, and other properties not included in this study.

A further study of water resistance utilizing asphalts obtained from several different crude oil sources would widen the basis of inference for conclusions concerning the effectiveness of anti-stripping measures.

VII. APPENDICES

APPENDIX A

Tables

## TABLE I

### PROPERTIES OF CRUSHED GLASS AGGREGATE







a Width/Thickness greater than 3.0

 $<sup>b</sup>$  Length/width greater than 3.0</sup>

# TABLE II

# PROPERTIES OF ASPHALT  $\!a$



a Furnished through courtesy of Shell Oil Company

# TABLE III

# GRADATIONS OF MIX DESIGNS



## TABLE lV

# RESULTS OF MARSHALL TESTS

First Mix Design Series

Asphalt Content (TWB)	Unit Weight (PCF)	Air Voids $(\%)$	<b>VMA</b> $(\% )$	Stability (LBS)	Flow IN) $\sqrt{100}$	
4.5 4.5 4.5 4.5 4.5 5.0 5.0 5.0 5.0 5.0 5.5 5.5 5.5 5.5 5.5 6.0 6.0 6.0 6.0 6.0 6.5 6.5 6.5 6.5 6.5 7.0 7.0 7.0 7.0	140.29 140.73 140.86 140.91 140.44 141.59 141.05 141.29 141.37 141.46 142.25 141.52 141.72 141.68 141.44 141.69 141.30 141.35 141.25 141.09 141.16 140.33 140.86 140.86 141.26 139.76 139.91 139.74 139.61	4.12 3.82 3.73 3.70 4.03 2.51 2.88 2.72 2.66 2.59 1.45 1.96 1.82 1.85 2.02 1.13 1.40 1.36 1.43 1.55 0.80 1.38 1.01 1.00 0.72 1.20 1.10 1.22 1.31	14.11 13.84 13.76 13.73 14.02 13.81 14.14 13.99 13.94 13.88 13.81 14.26 14.14 14.16 14.31 14.64 14.87 14.84 14.90 15.00 15.44 15.93 15.61 15.61 15.37 16.66 16.57 16.67 16.75	525 845 1050 760 920 600 1020 690 660 750 523 780 690 780 870 500 760 595 504 620 536 577 515 480 570 325 488 450 442	18 14 12 18 15.5 20 9 15 18 20 16 11 15 12 9 25 15 19 23 15.5 19 18 15 25 20 24 22 16 21	
7.0	140.21	0.88	16.39	442	21	

### TABLE V

#### MARSHALL DESIGN CRITERIA\*



\* Recommended by The Asphalt Institute for medium traffic (50-blow compaction)

### TABLE VI

# RESULTS OF MARSHALL TESTS Second Mix Design Series



### TABLE VII

### RESULTS OF SIEVE ANALYSES OF EXTRACTED AGGREGATES

## First Mix Design Series



# TABLE VII (cont.)

## RESULTS OF SIEVE ANALYSES OF EXTRACTED AGGREGATES

Second Mix Design Series



### TABLE VIII

# IMMERSION-COMPRESSION TEST RESULTS

# 1% ADDITIVE A



# TABLE VIII (continued)

# 4% ADDITIVE C



\* AVB = Aggregate Volume Basis

# TABLE VIII (continued)

# IMMERSION-COMPRESSION TEST RESULTS RETAINED STRENGTHS



# TABLE IX

# IMMERSION-COMPRESSION CRITERIAa



a<br>Goode, Joseph F., "Use of the Immersion-Compression Test in Evaluating and Designing Bituminous Paving Mixtures", ASTM STP No. 252, *p.* 113-125.

 $<sup>b</sup>$  Use as the desired value, plus or minus 0.3 percent.</sup>

#### TABLE X

### STATISTICAL SUMMARY OF MARSHALL TEST DATA SERIES I ANALYSIS OF VARIANCE TEST RESULTS

Hypothesis: The mean test property values for each asphalt content are equal.



\* If  $F_{\text{Calculated}}$  >  $F_{\text{Tabulated}}$ , reject hypothesis.

 $^{\textrm{\tiny{\textregistered}}}$  F $_{\textrm{\tiny{\text{T}abulated}}}$  is based on 0.05 significance level

## DUNCAN'S MULTIPLE RANGE TEST RESULTS



NOTE: Mean values which are underscored by a common line are not significantly different from each other. Mean values which are not underscored by a common line are significantly different from each other.

#### TABLE X (continued)

#### STATISTICAL SUMMARY OF MARSHALL TEST DATA SERIES II ANALYSIS OF VARIANCE TEST RESULTS

#### Hypothesis: The mean test property values for each asphalt content are equal.



\* If  $F_{\text{Calculated}}$  >  $F_{\text{Tabulated}}$ , reject hypothesis.

 $\texttt{a}$   $\texttt{F}$   $\texttt{Tabulated}$  is based on a 0.05 significance level.

<sup>b</sup>*nne* stability value was fitted due to a missing piece of data.

### DUNCAN'S MULTIPLE RANGE TEST RESULTS



NOTE: Mean values which are underscored by a common line are not significantly different from each other. Mean values which are not underscored by a common line are significantly different from each other.

# TABLE XI

### IMMERSION-COMPRESSION EXTRACTION DATA





APPENDIX B

 $\mathcal{L}^{\text{max}}_{\text{max}}$ 

Figures



Figure **l. Marshall** Test Property Curves-First **Trial** Mix



Figure 2. Marshall Test Property Curves-Second Trial Mix



Figure 3. Average Compressive Strengths for Immersion-Compression Tests



•





Figure 5. Effect of Differing Levels of Anti-Stripping Additives on Retained Strength

APPENDIX C

 $\label{eq:2.1} \frac{1}{\sqrt{2}}\int_{\mathbb{R}^3}\frac{1}{\sqrt{2}}\left(\frac{1}{\sqrt{2}}\right)^2\frac{1}{\sqrt{2}}\left(\frac{1}{\sqrt{2}}\right)^2\frac{1}{\sqrt{2}}\left(\frac{1}{\sqrt{2}}\right)^2\frac{1}{\sqrt{2}}\left(\frac{1}{\sqrt{2}}\right)^2.$ 

Statistical Analyses

 $\mathcal{L}^{\text{max}}_{\text{max}}$  and  $\mathcal{L}^{\text{max}}_{\text{max}}$ 

#### l. ANALYSIS OF VARIANCE

Data from the Marshall design series was analyzed with an analysis of variance technique using a randomized complete block design. These statistical procedures are described in detail in many standard statistical references. *A* randomized complete block design was chosen because day-to-day variations in laboratory temperature and humidity could give rise to variations in compaction. By insuring that one specimen representing each asphalt content was made each day, this source of variability was minimized in the analysis.

Following is the general procedure for analysis of variance of a randomized complete block design:



- 
- 1. Fill in table with data to be analyzed.
- 2. Compute row totals (A,B,G,D,E), column totals (a,b,c,d,e,f), and the grand total.

6o

3. Calculate:

- a) Correction Factor =  $(\text{grand total})^2/30$
- b) Total Sum of Squares = (Sum of the squares of each observation) - (Correction Factor)
- c) Treatment Sum of Squares =  $((a^2+b^2+c^2+d^2+e^2+f^2)/(number\ of$ rows)) - Correction Factor
- d) Block Sum of Squares =  $((A^{2}+B^{2}+C^{2}+D^{2}+E^{2})/(number \text{ of columns}))$  -Correction Factor
- e) Error Sum of Squares = Total Sum of Squares Treatment Sum of Squares - Block Sum of Squares
- 4. Enter values in this table:



- a Degrees of freedom for treatment and block equal the number of treatments minus one and the number of blocks minus one. Degree of freedom for error equals the product of the degrees of freedom of treatment and block.
- b Values are calculated in Step 3.
- c Value for each source obtained by dividing sum of squares for that source by its degree of freedom.
- d Value for each source obtained by dividing mean square for that source by the error mean square.

FTabulated values are obtained from a table of F values based on the level of significance desired. The level of significance shows the probability of making a Type I error (rejecting a hypothesis which should be accepted). The 0.05 level of significance is the probability of a Type I error occurring five times in 100 decisions. The abscissa of the F table represents the degree of freedom of the numerator of the F distribution, which in this case is the source being investigated (treatment or block), and the ordinate represents the degree of freedom of the denominator of the F distribution, which in this case is the error value.

If  $F_{\text{Calculated}}$  >  $F_{\text{Tabulated}}$ , the source being investigated has a significant effect on the test data. This is desirable for blocking since a greater  $F_{\text{Calculated}}$  indicates that a valid classification criterion was used in setting up the experiment.

Following is an example of the use of the randomized complete block design on one property of the Marshall test:

# RANDOMIZED COMPLETE BLOCK DESIGN

VMA - SERIES l

Treatment Block	4.5	5.0	5.5	6.0	6.5	7.0	Row Totals
$\mathbf M$	14.11	13.81	13.81	14.64	15.44	16.66	88.47
Tu	13.84	14.14	14.26	14.87	15.93	16.57	89.61
W	13.76	13.99	14.14	14.84	15.61	16.67	89.01
Th	13.73	13.94	14.16	14.90	15.61	16.75	89.09
$\mathbf{F}% _{0}$	14.02	13.88	14.31	15.00	15.37	16.39	88.97
Column Totals	69.46	69.76	70.68	74.25	77.96	83.04	445.15

$$
CF = \frac{(445.15)^2}{30} = 6605.28
$$
  
\n
$$
TSS = 6635.31 - 6605.28 = 30.03
$$
  
\n
$$
TRSS = \frac{33.173.28}{5} = 6634.66 - 6605.28 = 29.38
$$
  
\n
$$
BSS = \frac{39.632.36}{6} = 6605.39 - 6605.28 = 0.11
$$
  
\n
$$
ESS = 0.54
$$


## 2. DUNCAN MULTIPLE RANGE TEST

When the hypothesis to be tested is  $\mu_1 = \mu_2 = \mu_3$ , a significant F value does not provide complete information about the means. It is possible to make a statement that there are some significant differences among these means, but the immediate question of which means differ from others cannot be answered. The multiple range test is the best method of' determining which comparisons are significant.

To perform this test, three items of data are needed plus the necessary table of' "Significant Studentized Ranges":

- l. the means arranged in rank order,
- 2. the standard error of each mean  $(S_{\overline{x}})$ , and
- 3. the degrees of' freedom on which this standard error is based  $(N_2)$ .

The steps in the test are as follows:

- 1. the table for the significance level desired is entered at  $N_2$  = degrees of freedom for standard error from analysis of variance, and signficant ranges are extracted for sample size,  $p = 2, 3, 4, \ldots$
- 2. the significant ranges are then multiplied by the standard error of the mean,  $S_{\overline{x}}$ , to form the shortest significant ranges,
- 3. the appropriate shortest significant range is compared with each difference, and the difference is called significant if it exceeds the range, with one exception: No difference between two means can be declared significant if the two means are both contained in a set of means that has a non-significant range, and

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4. underscore, or mark by other ways, means that are not

significantly different.

An example showing how to follow the directions is as follows:

DUNCAN MULTIPLE RANGE TEST

VMA - SERIES l

Asphalt Content VMA Means 5.0 13.95 5.5 14.14 6.0 14.85 6.5 7.0 15.59 16.61 Let Content 4.5 5.0 5.5 6.0 6.5 7.0<br>
Standard Error of Treatment Mean =  $\sqrt{.027}/{5}$  = 0.073, for N<sub>2</sub>=20

SHORTEST SIGNIFICANT RANGES\*



There is no significant difference between VMA values for  $4.5$ and 5.0 percent and 5.0 and 5.5 percent asphalt content. All other comparisons show a significance difference.

\* NOTE: Mean values which are underscored by a common line are not significantly different from each other. Mean values which are not underscored by a common line are significantly different from each other.

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## IX. VITA

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