# IOWA STATE UNIVERSITY Digital Repository

**Retrospective Theses and Dissertations** 

Iowa State University Capstones, Theses and Dissertations

1960

# Influence of some Iowa aggregates on the durability of asphaltic concrete mixes

The Ngoc Phan Iowa State University

Follow this and additional works at: https://lib.dr.iastate.edu/rtd Part of the <u>Civil Engineering Commons</u>

## **Recommended** Citation

Phan, The Ngoc, "Influence of some Iowa aggregates on the durability of asphaltic concrete mixes " (1960). *Retrospective Theses and Dissertations*. 2389. https://lib.dr.iastate.edu/rtd/2389

This Dissertation is brought to you for free and open access by the Iowa State University Capstones, Theses and Dissertations at Iowa State University Digital Repository. It has been accepted for inclusion in Retrospective Theses and Dissertations by an authorized administrator of Iowa State University Digital Repository. For more information, please contact digrep@iastate.edu.



This dissertation has been microfilmed exactly as received

Mic 61-468

# PHAN, The Ngoc. INFLUENCE OF SOME IOWA AGGREGATES ON THE DURABILITY OF ASPHALTIC CONCRETE MIXES.

Iowa State University of Science and Technology Ph.D., 1960 Engineering, civil

University Microfilms, Inc., Ann Arbor, Michigan

# INFLUENCE OF SOME IOWA AGGREGATES ON THE DURABILITY OF ASPHALTIC CONCRETE MIXES

Ъy

The Ngoc Phan

A Dissertation Submitted to the Graduate Faculty in Partial Fulfillment of The Requirements for the Degree of DOCTOR OF PHILOSOPHY

Major Subject: Highway Engineering

Approved:

Signature was redacted for privacy.

## In Charge of Major Work

Signature was redacted for privacy.

## Head of Mafor Department

Signature was redacted for privacy.

# Dean of Graduate College

Iowa State University Of Science and Technology Ames, Iowa

# TABLE OF CONTENTS

INTRODUCTION 1
PURPOSE AND SCOPE OF INVESTIGATION
REVIEW OF PREVIOUS WORK
Types of Limestones
PROCEDURE
Physical Properties of Binders and Aggregates33Bituminous binders33Mineral aggregates35Resistance to Degradation43Absorptive Characteristics of Aggregates45Theory46Resistance to Stripping by Water53
RESULTS AND DISCUSSION
Physical Properties of Bituminous Binders
CONCLUSION
LITERATURE CITED111
ACKNOWLEDGMENT
APPENDIX

.

Page

#### INTRODUCTION

Since the end of World War II, the growth of highway transportation in the United States has exceeded the expectations of its most enthusiastic advocates. This phenomenal expansion brought with it problems whose solutions require much ingenuity on the part of the highway engineers.

As the traveling public grows more sophisticated, the demand has shifted from greater mileage to higher quality roads. The increased number and heavier weights of trucks, the scarcity of building materials, further complicate the situation.

The road builders are therefore turning to the research workers with the question: "How to build better and longer lasting roads with the available materials." The answer to this query is crucial, since the highway planners must provide a reasonable benefit-cost ratio to justify the financing of their projects. This benefit-cost ratic is usually based on the assumption that a high-type pavement will last from twenty to thirty years. This assumption is correct on the whole, but there have been too many exceptions to disturb the mind of the conscientious engineers.

Proud of their accomplishments in the past, expecting even greater successes in the future, the highway engineers

feel they are on the threshold of a new era, in which road building will become a science, in fact as well as in name.

Much remains to be done to turn a dream into reality, but a knowledge of the factors which contribute to the durability of flexible pavements would be a great step toward reaching that cherished goal.

## PURPOSE AND SCOPE OF INVESTIGATION

In July 1957, the Bituminous Research Laboratory of the Iowa Engineering Experiment Station started research project 346-S, sponsored by the Iowa Highway Research Board.

This project studies the effects of certain Iowa limestones on the durability of the bituminous concrete pavements in which they were used.

In order to correlate the results of laboratory tests with field performance, four sections of U.S. highways, designated A, B, C, and D respectively, were chosen.

The first three were laid during the summer of 1957 and the fourth section in the spring of 1958.

All four projects consisted of a bituminous concrete resurfacing of an old concrete pavement. However, in the case of Project D, welded wire-fabric reinforcement was placed in the binder course. Figure 1 shows the locations of the four projects; Table 1 gives the dates of construction and traffic data and Table 2 the weather data.

The behavior of an asphaltic concrete pavement depends upon:

- a) The quality of the bituminous binder
- b) The quality of the mineral aggregates
- c) The affinity between the binder and the aggregates.

	Date of construction	Average daily traffic, 1959	Truck traffic % of total traffic
Project A	July 1957	1,960	11.0
Project B	June 1957	2,490	12.0
Project C	August 1957	1,755	15.0
Project D	April 1958	4,335	16.0

Table 1. Dates of construction and traffic data<sup>a</sup>

<sup>a</sup>Gunnerson, Howard. Safety and Traffic Department. Iowa State Highway Commission, Ames, Iowa. Data from traffic survey. Private Communication. 1959.

Table 2. Climatological data<sup>a</sup>

Y	I Zear	Project A	Project B	Project C	Project D	
Average monthly pre-	1958	27.05	26.60	22.82	25.56	
cipitation, in. <sup>5</sup>	1959	31.07	33.11	32.70	33.73	
No. of days with	1958	1 <b>56</b>	151	153	151	
lows < 32F	1959	164	153	133	151	
No. of days with	1958	13	16	14	8	
highs >90F	1959	20	30	28	16	

<sup>a</sup>Data taken from (46)

<sup>b</sup>Precipitation was computed on the basis of 10 in. snow equals 1 in. rain

Project 346-S deals with all these factors.

This report concentrates more specifically on the contribution of the aggregates to the durability of the bituminous

Figure 1. Map of Iowa showing the locations of the four projects



Figure 2. Geological map of Iowa showing the sources of the aggregates used in the investigation



PRECAMBRIAN

6b

pavement by examining:

- a) their resistance to abrasion,
- b) their absorptive characteristics as a factor in the hardening of the binder, and
- c) their adhesion to the respective asphalt cements in the presence of water.

The physical properties of the asphalts and aggregates and the mineralogical composition of the latter must also be known.

Although the subgrade and base play an important role in the behavior of a flexible pavement, they are outside the scope of this investigation. In this paper, the term "pavement" refers exclusively to the asphaltic concrete surface course.

All the materials in these projects have passed the inspection of the Iowa Highway Commission as satisfactory for the particular jobs. The highway sections under consideration are still in good condition and the maintenance on these roads has been reported as routine.

## REVIEW OF PREVIOUS WORK

Types of Limestones

The word "limestone" is a general term for that class of rocks which contain at least fifty percent of the carbonates of calcium or magnesium.

A classification of limestones based on origin would read (35):

1. Accretionary or autochthonous limestone

2. Clastic or allochthonous limestone

- 3. Chemical limestone
- 4. Dolomitized limestone.

Pettijohn (35) devides limestones into five subclasses, according to the ratio of calcite to dolomite:

Table 3. Carbonate rock terminology<sup>a</sup>

Туре	Calcite	% dolomite
Limestone	95-100	0-5
Magnesian limestone	90-95	5-10
Dolcmitic limestone	50-90	10-50
Calcitic dolomite	10-50	50-90
Dolomite	0-10	90-100

<sup>a</sup>Data from (35)

The rocks of Iowa (19) belong to only four kinds: calcareous, arenaceous, argillaceous or carbonaceous. The first class comprises limestones and dolomites and are dominant in the state.

Only six different elements appear in these four kinds of rocks. Three of these are metals: calcium, magnesium and aluminum. The other three are metalloids: oxygen, carbon and silicon.

The calcareous rocks of Iowa have the following average chemical composition:

Rocks and corre-	Elements						-
sponding minerals	Metalloids			Metals			
	0 <sub>2</sub>	Ca	Si	Ca	Mg	A1	
Calcareous rocks				•			
Calcite	48	12		40			
Dolomite	52.2	13		21.8	13		

Table 4. Chemical composition of calcareous rocks

This difference in oxygen content between calcite and dolomite may prove important in the rate of oxidation of the asphaltic binder.

The calcite-dolomite ratio in limestones can be determined by X-ray diffraction, differential thermal, petrographic or chemical analyses. Prominent among the latter are the more or less simple procedures using ethylenediaminetetraacetic acid (EDTA) as a reagent (47, 32, 2).

Emphasis has been placed, and rightly so, on the quality of the asphaltic binder whenever the subject of durability of bituminous pavements is discussed. However, the mineral aggregates undoubtedly play an important role in this phase of design.

There are three main areas where the influence of the aggregates on the performance of the asphaltic concrete mixture is felt:

- 1. The intrinsic toughness of the aggregates
- 2. Their absorptive characteristics
- 3. Their adhesive properties in relation to bituminous binders.

#### Resistance to Degradation

The first requirement of a good aggregate for road construction is its ability to withstand the action of traffic without excessive abrasion and breakage. This has been recognized and standard tests such as the Deval and Los Angeles procedures have been devised to evaluate that quality (1).

Melville (27) lists the factors causing aggregate degradation as follows:

Chemical actions	Physical actions
Oxidation and reduction	Wear
Hydration and dehydration	Impact
Carbonation and decarbonation	Splitting
Silicification and desilicification	Abrasion
Sulphidation and desulphidation	Fatigue
Solution and precipitation	Shearing
	Attrition
	Crushing

A poor aggregate is more likely to disintegrate under the physical actions first.

The standard tests rely on impact and are concerned only with the coarse aggregates. In the same vein some fundamental research on the problem of comminution have been conducted by the petroleum industry.

Most theories on comminution are based on the empirical proposition that the energy required to make a small change in the size of an object is proportional to the size change and inversely proportional to the object size to some power n. In mathematical form, this proposition would appear as: (5)

# $dE = -Cdx/x^n$ .

The problem consists in assigning a value to the exponent n.

Rittinger (5) postulated that the energy required for size reduction of a solid would be proportional to the new surface area created during the size reduction. He thus gave to n the numerical value 2.

Kick (5) proposed the theory that equivalent amount of energy should result in equivalent geometrical changes in the sizes of the pieces of a solid. For him, n should be equal to 1.

Rittinger's theory refers to the energy absorbed in the rupture of chemical and physical bonds during fracture of the particles, and Kick's law to the energy required to deform the particles to its elastic limit (15).

Neither of the above hypotheses has received general acceptance.

Bond tried to connect the two previous theories and postulated that "the total work, useful in breakage, which has been applied to a stated weight of homogeneous material is inversely proportional to the square root of the diameter of the product particles" (15). In other words, he assigned to the exponent n the value 1.5.

According to Holmes (15), the work-input to a sizereduction operation is consumed in three separate ways:

- 1. In effecting elastic deformation
- 2. In effecting plastic deformation, and
- 3. In producing new surface.

For most naturally occurring rocks the elastic limit and final yield point are so close together that, for practical purposes, work done in plastic deformation may be neglected. Furthermore, the proportion of the total input energy converted into surface energy is extremely small. This enabled Holmes to use Hooke's law in developing his theory.

Charles (5) also derived a general Energy-Size reduction relationship based on the size-distribution curves.

The only limitation to his method is that the shapes of these curves must remain constant and suffer only a lateral translation on a size-distribution graph with change in energy input.

This stipulation is not as severe as it at first seems, for the results from comminution tests show that for a given material and for a given means of size reduction the resulting size distribution curve generally changes little in shape and is only translated bodily by changes in energy input.

Turner and Wilson (44) found that the susceptibility of an aggregate to degradation increases as the size of the particles decreases. This could be due to the sharply increasing surface area of the particles.

In their study, they used a compactor to simulate the effect of wheel loads. Their results indicate that the outer weathered surface of the particles contribute more heavily to the plasticity of the fines than do the inside materials. The effect of these plastic fines in a compacted roadbed is that of a lubricant, allowing shifting and movement to take place under wheel loads.

Melville (27), using the ball-mill tests, found that high sample wear could usually be correlated to poor field performance and, in the case where an aggregate of good field performance did show high sample wear, that the fines were non-plastic.

Knight (22) used a static-load compaction procedure to study the behavior of five aggregates--norite, dolerite, basalt, dolomite and quartzite--under traffic action.

Successive crushing tests on the chosen aggregates show that final gradings were reached, after which there was little further size reduction under load and only a small increase in bulk density. The final grading was shown to be influenced by the original grading and the type of stone used.

Absorption Characteristics of Aggregates

The importance of the absorption characteristics of aggregates in the design of bituminous mixes has been pointed out by Nevitt and Krchma (30), among others.

The primary effect of absorption is to deplete the binder film. The binder is absorbed into the aggregate leaving thinner films more susceptible to weathering and water action (7).

There exist standard procedures to determine the absorption of water by aggregates (1). In 1942, Hveem came out with his Centrifuge Kerosene Equivalent (CKE) test (18). Since its inception, this method has been extensively correlated with field experience and is being used as a part of the design of bituminous mixtures by some Western states.

Donaldson, Loomis and Krchma (7) proposed some modifications to the standard CKE method, notably by increasing the soaking time to thirty minutes and by testing a non-absorptive aggregate of the same gradation as the unknown aggregate, for purposes of comparison.

Question may be raised about the validity of predicting the amount of asphalt absorption from test results obtained by using water or kerosene. The question seems to revolve around the viscosity of the liquid in question. Lettier and

 $QV^a = b$ 

where

- Q = m1 of oil absorbed per 100 gms of aggregate at constant soaking time
- V = viscosity in centistokes at soaking temperatures
- a = constant related to the shape and general nature of the voids in the aggregates
- b = total quantity of voids.

Absorption enters the aggregates through their interconnecting pores. Not a great deal is found in the highway engineering literature about the determination of porosity and pore-size distribution. Fortunately the oil industry again comes to the rescue with papers by Leverett (25), Hassler, Brunner and Deahl (14), among others. Purcell (36) described an apparatus using mercury to measure capillary pressures and from these, the permeability of porous solids can be calculated. Ritter and Drake (38) also presented a method for determining the pore-size distribution in porous materials from the data given by a pressure porosimeter.

## Adhesive Characteristics of Aggregates

In general, dense-graded asphaltic concrete offers good resistance to moisture, provided the mix is properly designed and has high density.

Three conditions (31) are found to contribute to the failure of pavements subjected to excessive moisture:

- a) Excessive clay content in the aggregate fraction passing the No. 200 sieve,
- b) Low bitumen content, and
- c) High voids in the pavement due to lack of proper compaction.

It is common practice to speak of a hydrophilic or hydrophobic aggregate. This classification is misleading, because both the aggregate and the binder influence the behavior of the mixture in the presence of water.

Rice (37) enumerates the following properties of aggregates as affecting the adhesion of bituminous materials in the presence of water: surface texture, surface coatings, particle size and surface area, porosity and absorption, chemical reactivity and surface energy.

Surface texture is determined by the size and hardness of the individual grains, cementitious material and pore structure. There is no general agreement, although some attempts have been made to classify rocks according to surface texture (23).

Surface coatings: coatings may be composed of clay, silt, calcium carbonate, iron oxides, opal, gypsum, manganiferous

substances, and soluble phosphates (3). For practical purposes, the most important coating is water. Water films may be held very tightly to the surface (43), and possibly in a crystalline, rather than a liquid form (12).

Particle size and surface area: An excess of fine particles may require more binder than is compatible for stability (42). The high surface area of the small particles means greater amount of surface energy resulting from broken internal bonding forces. For a given particle size, the surface area depends upon the surface texture, the coatings present, and the shape of the particle.

Porosity and absorption: The amount of bituminous material absorbed depends upon the total volume of permeable pore space, the size of the pore openings, the viscosity and surface tension of the bituminous material, and the time during which the bituminous material is fluid.

Chemical reactivity: It is known that carbonate rocks are subject to attack by acid solutions, but how important is the reactivity with bituminous materials has not been definitely established, in spite of suggestions by several investigators (16, 41). Ebberts (8) has suggested that limestones may lose some carbon dioxide when heated, resulting in the formation of a film of calcium oxide on the surface. However the temperature of dissociation of calcium carbonate

is about 1650°F.

Surface energy: The surface energy of minerals has been attributed to a) broken coordination bonds of the crystal lattice (43), b) electronic or ionic forces (16), c) electrical charges (48), d) molecular attractions (29), e) the polar nature of the minerals (16), f) adsorbed ions (43, 48), g) the bonding action of adsorbed polar molecules (12).

Three main theories have been advanced to explain the water-resistance of bituminous-coated aggregates:

1. The chemical reaction concept which states that the acidic components of the bituminous material react with basic minerals to form water-insoluble compounds.

2. The mechanical concept which emphasizes the role of surface roughness and mechanical adhesion.

3. The surface energy concept which concerns the adhesion resulting from the interfacial energy, relationships at the aggregate-bitumen-water-air interface.

Chemical reaction concept: The importance of the acidic or basic composition of the aggregate seem to be supported by the observations that acidic rocks tend to strip more readily than basic rocks (13).

Mechanical theory: According to this concept, the composition of the rock, mineral or chemical, is important only

in so far as it affects the surface texture. Particles with smooth surfaces are more easily wetted and require less bituminous material than particles with rough surface (16). Angular particles with sharp edges may be more difficult to coat uniformly and the thin films at the edges may be less water resistant (42). Porous aggregates, once wetted with asphalt show better adhesion due in part to a mechanical interlock which mobilizes cohesive forces (16). Pauls (33) states that absorptive rocks, while hard to coat, retain bituminous films very well. However, Tyler (45) found that there was no correlation between porosity or absorptive capacity of an aggregate and the amount of stripping resistance. Pauls and Rex (34) found that clay as a coating was more deleterious than clay as a filler.

Surface energy concept: It is the most widely accepted, but most complex and least understood. Three possibilities occur when a liquid and a solid are brought into contact (16):

1. The liquid may neither spread on nor wet the surface of the solid (mercury and glass)

2. The liquid may cover the surface without wetting. This happens when there is a layer of water or dust between the stone and the asphalt.

3. The liquid may spread on and wet the surface. This is the desirable result that the asphalt technologist tries to obtain.

For a liquid to spread readily on a solid, the surface tension of the liquid should be less than that of the solid. High surface tension of a liquid works against spreading (17, 17). Conversely, the higher the surface tension of the solid, the greater the tendency for the solid to draw the liquid over its surface (41). When a liquid spreads over and wets a solid there is a reduction in the surface energy of the separate phases and a creation of a new interfacial relationship.

Many investigators have used various approaches to the problem of stripping by water. Some of them require highly technical skill, such as measurement of surface tension by bubble methods, of wetting by determination of contact angles, of adhesion tension by capillary rise and of interfacial tension by volume determination of free-settling powders.

The test methods could be briefly classified as follows (10):

- 1. Coating or mixing tests in the presence of water.
- 2. Static immersion tests.
- 3. Immersion tests employing agitation (wash tests).
- Water displacement tests employing hot or boiling water with or without electrolytes.
- 5. Tests which measure surface activity or interfacial tension.

- Tests which measure change in volume on exposure to water (swell tests).
- 7. Tests which measure strength or strength properties before and after water exposure.
- 8. Simulated traffic tests or track tests.

The first four tests can be considered qualitative, and the last four quantitative. However this classification is not rigid. Plate 1. View of sawed section of Project A pavement core

Plate 2. View of sawed section of Project B pavement core





Plate 3. View of sawed section of Project C pavement core

Plate 4. View of sawed section of Project D pavement core





Plate 5. Close-up view of sawed section of Project A pavement core

Plate 6. Close-up view of sawed section of Project D pavement core

•



.



Plate 7. Close-up view of sawed section of Project B pavement core

.


Plate 8. General view of the asphalt-reclaiming apparatus

Plate 9. The sliding-plate microviscometer in operation



• •.



.

### PROCEDURE

Physical Properties of Binders and Aggregates

### Bituminous binders

All the asphalt cements used were 85-100 penetration grade. The non-standard tests are described as follows:

a) The "Thin Film Oven" test was devised by the Bureau of Public Roads in 1941 (26). In this test, asphalt was spread in a standard container in 1/8-in. thick film and heated in a prescribed oven for five hours at  $325^{\circ}$ F. Routine tests, such as softening point, penetration, ductility, were then run on the residues from this test.

b) The asphaltene content, expressed as a percentage of the total asphalt used, was obtained by the selective solvent method proposed by Csanyi and Fung (6). Skelly F was used as solvent. The extraction time in the soxhlet extractors was maintained for eight hours.

c) A Hallikainen "Sliding Plate Microviscometer" was used to determine the viscosity of all asphalts. The details of the procedure are contained in a paper by Griffin, Miles and Penther (11). After plotting the values obtained from the test on a log-log graph paper, the viscosity corresponding to a shearing rate of  $5 \times 10^{-2} \text{ sec}^{-1}$  was reported in poises. The thicknesses of the films used in this study varied from 20 to 45 microns.

The asphalt cement was extracted from the asphaltic concrete mixes by the standard A.S.T.M. Rotarex procedure (A.S. T.M. D 1097-54T) with benzene as the solvent. These mixes included both laboratory samples and road cores.

The colloidal mineral matter was separated from the asphalt-benzene solution by further centrifugation at 400 G for ten minutes.

The Abson method (A.S.T.M. D 762-49), with some modifications, was used to reclaim the asphalt in the following manner:

A three-necked 500-ml pyrex flask constituted the main element of the apparatus. The right neck admitted a thermometer, the left, a connection between flask and condenser and the middle neck provided entrance for the gas dispersion tube.

In the early stages of the distillation, a cork closed the central neck. A thermostat, connected to an electric heating mantle which fitted the lower part of the flask, enabled the operator to maintain the contents of the flask at a temperature of 190  $\pm$  10° F. When the rate of condensation of benzene decreased to about 4 drops per minute, carbon dioxide gas was admitted through the middle neck by means of a dispersion tube. The application of gas, at a rate of 1000 ml/ min., was continued until the benzene came out at the rate of

one drop per minute.

At ten minutes and thirty minutes after this end point was reached, samples of the reclaimed asphalt were collected.

Viscosity and softening point tests were made on this material to determine the changes following various treatments in the laboratory or undergone in the field under the action of traffic and weathering.

## Mineral aggregates

A geological map of Iowa (20) shows that the limestones used in projects A, B and D are Mississippian in age, those of Project C are Pennsylvanian.

<u>Mineral composition</u> Two methods were used to determine the principal mineral components of the aggregates:

1. X-ray diffraction method. In spite of its qualitative character, it proved to be adequate for the purpose of this investigation. Although both coarse and fine aggregates were analyzed, only the results pertaining to the limestones will be discussed.

In the analysis, a General Electric XRD-5 X-R Diffraction Unit was used. The aggregates were powdered with a mortar and pestle until the powder passed through a No. 200 sieve. Care was taken to obtain rocks from different bins and to use a

representative sample of the combined powder for the test.

Since the determination of clay minerals was not desired, the glycol treatment served only to hold the powder together in the flat aluminum sample-holder. The accompanying graphs are reproductions of the original diffraction patterns as obtained on the recording papers.

2. Determination of calcite and dolomite: Since limestones are carbonate rocks, the respective percentages of calcite and dolomite in the aggregates are of special interest.

The experimental procedure followed was that described by Bisque (2), with a few minor modifications.

<u>Principle</u> A standard solution of EDTA (Ethylenediaminetetraacetic acid) and visual indicators are used to determine the amount of calcium and magnesium in limestones.

Since these two metal ions react quantitatively with EDTA under the same conditions, the sum of calcium and magnesium can be titrated in a small amount of the sample. By titrating another portion of the same sample at a higher pH (12-14), the amount of calcium is obtained, the magnesium having been rendered unreactive at this higher pH level. The amount of magnesium is then the difference between the two determinations.

In the titration of calcium and magnesium the interfering ions which may be encountered are: iron, copper, aluminum, manganese, cobalt, nickel and zinc.

The addition of cyanide serves to complex copper, cobalt nickel and zinc ions to prevent them from reacting with EDTA. Iron and manganese are reduced to their lower level states by hydroxylamine hydrochloride and thereby prevented from interfering. Aluminum must be complexed by the addition of sodium tartrate.

<u>Sample treatment</u> The stone particles, selected at random, were reduced in size to pass the No. 100 sieve with a mortar and pestle. The powder was dried to constant weight in an oven at  $100^{\circ}$  C. After cooling to room temperature in a dessicator, 1.00 gm of the sample was placed in a 400-m1 beaker; 150 ml of 3N hydrochloric acid was slowly added to the beaker and the solution was held at 50-60° C for one-half hour. The solution was then transferred directly to a 1000-ml volumetric flask, cooled and diluted to volume.

Procedure for the determination of calcium and magnesium 10 ml of the solution were pipetted into a 250-ml widemouthed Erlenmeyer flask. The following ingredients were then added:

10 ml of the ammonium-ammonium chloride buffer solution to raise the pH of the solution;

5 ml of the 10 percent hydroxylamine hydrochloride solution;

10 ml of the potassium cyanide solution;

several crystals of sodium tartrate and a small pinch of the F241 indicator (Eriochrome Black T).

The contents of the flask were mixed thoroughly with a magnetic stirrer and titrated with the 0.01 M standard solution of EDTA. The end point was indicated by a change from a wine-red to a blue color which did not fade.

The number of milliliters required for this titration multiplied by 0.01 gave the number of millimoles (mM) of calcium and magnesium in 10 ml of solution.

Procedure for the determination of calcium in the presence of magnesium

10 m1 of the solution were placed in a 250-m1 Erlenmeyer flask. The following ingredients were then added:

3-4 ml of the potassium hydroxide buffer solution; 5 ml of the hydroxylamine hydrochloride solution; 10 ml of the potassium cyanide solution;

several crystals of sodium tartrate, and about 0.1 gm of the murexide indicator mixture or "Calver II".

<u>Preparation of reagents</u> Buffer solution for the titration of calcium plus magnesium: 68 gms of ammonium chloride were placed in a 1000-ml measuring flask; 570 ml of concen-

trated ammonium hydroxide were added and the solution was diluted to volume with distilled water.

Hydroxylamine-hydrochloride solution: 10 gm of hydroxylamine hydrochloride were dissolved per 100 ml of solution.

Potassium cyanide solution: 2.5 gm of potassium cyanide were dissolved per 100 ml of solution.

Indicator for the titration of calcium plus magnesium: 150 gms of sodium chloride were placed in a mortar and 0.5 gm of Eriochrome Black T added. The solids were ground with a pestle until an homogeneous mixture was obtained. A small pinch was used in the titration.

Indicator for the titration of calcium in the presence of magnesium: the solid indicator "Calver II", sold by the Hach Chemical Co. of Ames, Iowa, was used.

Standard calcium solution: 1.00 gm of pure calcium carbonate was transferred to a 1-liter volumetric flask with a powder funnel. The powder funnel and the neck of the flask were rinsed with distilled water.

250 ml of 0.10 N hydrochloric acid were added and the contents swirled until solution was complete; 40 ml of 0.10 N sodium hydroxide were added, the contents of the flask were then cooled to room temperature and diluted to the mark.

Standard EDTA solution: all the quantities given below apply to the preparation of one liter of solution.

3.9 gms of disodium salt of EDTA were weighed into a storage bottle and diluted to one-liter volume with distilled water. The strength of the EDTA stock solution was adjusted by diluting with water or, if necessary, by adding more EDTA, until 10.0 ml of the solution were required to titrate 10.0 ml of the standard calcium solution to the blue end point. The EDTA stock solution was then 0.1 M and suitable for the determination of calcium and magnesium as outlined. (M stands for molarity.)

Calculations:

- A = millimoles of Ca + Mg = ml used to titrate part 1 of sample multiplied by 0.01.
- B = millimoles of Ca = ml used to titrate part 2 of sample multiplied by 0.01.

C = millimoles of Mg = A - B.

Since dolomite contains both calcium and magnesium, a slight difficulty arises. However, if a rock were pure dolomite, the ratio Ca/Mg would be equal to that of their respective molecular weights:

Weight of Mg = 
$$\frac{40.08}{24.32}$$
 = 1.648

Total weight of calcium:

$$D = \frac{(B)(Mol. wt. of Ca)}{(1000)(0.01)} gm/gm of sample$$

Total weight of dolomite:

$$E = \frac{(C) (Mol. wt. of Mg)}{(1000)(0.01)} gm/gm of sample$$

Weight of Ca in dolomite = F = (E)(1.648)Weight of Ca in calcite = G = (D) - (F)Percent of calcite =  $G/D \times 100$ 

Percent of dolomite =  $F/D \times 100$ 

These two percentages should add up to one hundred, because of the assumption that calcite and dolomite are the only components of limestones.

<u>Density</u> The bulk specific gravity was checked by the standard A.A.S.H. O-T 96-49 method. The apparent specific gravity was obtained by means of the powder (true) specific gravity instead of the standard A.A.S.H.O method, because the former gave better reproducibility. The following description of the procedure is reproduced from Rush (39):

The specific gravity is computed from the measured weight and the volume displacement of the sample.

The volume displacement of the sample is determined by the use of a volumetric flask having 100-ml and 110-ml marks.

The sample used is powdered to pass the No. 100 mesh U.S. Standard sieve.

The weighed sample is transferred to the volumetric flask and 50 ml of 50% acetone-water solution is added from a 50-ml pipette, followed by agitation of the flask for 60 seconds to

eliminate any trapped air and to insure complete wetting of the sample. A 50% acetone-water solution is selected because of its high wetting ability.

Another 50 ml of acetone solution is then added from the pipette. It is necessary to use a sample of about 25 grams so that the sample volume plus 100 ml of acetone solution will not exceed 110 ml and the level of the fluid will remain between the two calibration marks.

With the aid of a 25-ml burette calibrated to 0.05 ml units, distilled water is added to the volumetric flask so that the volume of the sample plus the acetone solution is increased to 110 ml. The amount of liquid added is recorded.

The powder specific gravity is computed by the formula: Powder specific = (Sample weight in grams) gravity Flask volume -[(100 ml) + (ml added from burette)]

<u>Texture and shape</u> Because of their important role in the stability of the bituminous mixes, the texture and shape of the coarse aggregates were examined visually. The terminology used to describe them is borrowed from Field (9) and Pettijohn (35).

#### Resistance to Degradation

The standard Los Angeles Abrasion Test (A.A.S.H.O -T 96-49) is limited to the coarse aggregates, and for this reason, may not give a **true** picture of the behavior of the mix under actual service conditions.

The two factors that mitigate the crushing effect of loading on the coarse particles are believed to be: a) the cushion provided by the fine aggregates and b) the lubrication due to the binder, which permits some reorientation of the coarse aggregates.

To examine these two unknowns, the following series of tests were run.

a. Aggregates alone: For each project, three samples of aggregates were made up, using the same gradation, which is the median of the two limiting gradations, as set forth in the Iowa Specifications for Asphaltic Concrete Mixes, Type A. with 3/4" maximum size aggregates (21).

These aggregate blends were then compacted in the Hveem's Kneading Compactor, following the same procedure and using the same number of blows as in the case of bituminous mixes.

A sieve analysis was run on the products of compaction and a "degradation ratio" defined as:

b. Aggregates and binders: Using a similar gradation as in part (a), the following study was made on: 1) the effect of the Hveem's Kneading, the Marshall's Drop-Hammer and the Double-Plunger Static methods of compaction, and 2) the effect of binder contents on the degradation of the aggregates.

In order to characterize the latter, a new quantity, called the "filler-bitumen factor", was introduced. It is defined as:

F-B Factor =  $\frac{\text{Ratio of \% filler to \% bitumen in mix after comp.}}{\text{Ratio of \% filler to \% bitumen in mix before comp.}}$ the term "filler" referring to the portion of the aggregates passing the No. 200 sieve.

The Hveem and Marshall compaction methods are described in the Asphalt Institute's Manual Series No. 2 (28).

In the Double-Plunger Static compaction, the steel plates between the mold and the base plates are removed when the load reaches 1,000 lbs. Loading continues at the same constant rate (0.05 in./min.) until the load reaches 3,000 psi; it is maintained at that level for three minutes and released slowly. c. Road cores: None of the compaction methods considered can reproduce the compaction by rollers and other construction equipment, and the consolidation induced by fast moving traffic. In order to study the behavior of aggregates in the mixes under actual wheel loads, cores 4" in diameter were cut from the four pavements, using a Howe-Stimpson HS-12 portable rig. These cores have been under traffic for different periods of time and they were taken from both under and between the traffic lanes.

Absorptive Characteristics of the Aggregates

All aggregates are porous by nature, some to a greater extent than others. At the usual mixing temperature, asphalt cements behave more or less as liquids and may penetrate into the larger pores of the aggregates. Due to their lower molecular weights and viscosities, the resins and oily constituents of asphalt would probably enter these pores first, leaving the binder on the surface of the aggregates richer in asphaltenes and, as a consequence, harder. However if selective absorption does not occur, then any appreciable amount of absorption would result in thinner asphalt films on the aggregate particles.

It has been generally agreed, in the United States at least, that a very hard asphalt and/or a very thin film of binder on the aggregates would shorten the life of the pavement

in which it occurs.

Thus, not only the amount but also the size-distribution of the pores are of interest.

The total porosity of the limestones was calculated from the equation:

Percent porosity = 
$$100 - \frac{Bulk specific gravity}{Powder specific gravity} \times 100$$
.

The pore-size distribution of the stone particles was determined by forcing mercury under pressure into the evacuated pores of the solids, using an apparatus described by Purcell (36). This method applies only to the determination of interconnecting "macropores", e.g., to pores larger than 100 angstroms.

## Theory

The system involves a porous solid and a single nonwetting fluid (mercury) which forms a contact angle greater than  $90^{\circ}$  against the solid. The action of the surface forces involved opposes the entrance of the liquid into the solid and pressure must be applied to the liquid to cause penetration of the pores of the solid.

The relation (38) giving the pressure required to force liquid into a pore of given size is

$$pr = -2 \boldsymbol{\sigma} \cos \boldsymbol{\Theta} \qquad 1$$

where p is the pressure applied, r the pore radius,  $\delta$  the surface tension of mercury, and  $\Theta$  the contact angle of mercury against the solid.

This means that a porous material, with pores larger than 100 angstroms in diameter, under zero pressure will absorb none of any non-wetting liquid in which it is immersed. When the pressure is raised to some finite value, the liquid will penetrate and fill all pores having radii greater than that calculated from Equation 1. As the pressure is increased, the amount of liquid absorbed increases at a rate proportional to the differential pore volume due to pores of size corresponding to the instantaneous pressure. Thus, a given pore-size distribution gives rise to a unique pressuring curve.

<u>Apparatus and testing procedure</u> The essential components of the apparatus are a mercury displacement pump, a sample holder and a manifold system, wherein the nitrogen pressure may be varied from high vacuum to about 2000 psi, gauge.

The mercury pump consists of a piston-cylinder arrangement, the former being moved by means of an accurately machined screw, the pitch of which is such that one turn of the driving mechanism moves the piston through a distance sufficient to displace one cubic centimeter. The volume of liquid displaced from the pump is determined by successive readings of

the scale and vernier which is attached to the hub of the hand wheel.

The sample holder consists of two parts, both of which carry a lucite window which is cemented into the body of the holder and held rigidly in place by bushings. The displacement pump is connected to the sample holder and manifold by means of diametrical conduits through the two luctie plugs. Reference marks are incorporated in these conduits at about the midway point of the lucite windows and may be viewed through the openings in the supporting bushings.

In operation, about 22 grams of coarse aggregates particles, which have been dried for 24 hours in a vacuum oven at  $140^{\circ}$  C, was placed in the cavity of the sample holder. The top portion of the sample holder was positioned and the two parts brought together by a make-up nut. A suitable gasket made the seal pressure tight.

With the mercury level somewhat below the reference line of the lower lucite window, a vacuum was drawn on the system until an absolute pressure of 0.005 mm. of mercury, or less, was registered by the McLeod gauge. The mercury level was then accurately positioned at the lower reference mark by advancing the piston of the displacement pump. The scales attached to the volumetric pump were set at zero following which the piston was further advanced until the mercury meniscus

reached the reference mark in the top lucite window. At this point a scale reading was made which indicated the amount of mercury required to fill the cell with the sample in place. This quantity was subtracted from the known volume of the sample holder (between the reference marks) to provide a measure of the bulk volume of the sample under test.

The vacuum pump was isolated from the manifold and gas admitted into the system in increments, thereby increasing the pressure on the mercury surrounding the sample. The entrance of mercury into the pores of the aggregates was indicated by a recession of the mercury-gas interface from the upper reference line, and the degree of penetration was determined by advancing the displacement pump piston until the mercury meniscus returned to this reference mark.

The procedure of alternately building up the pressure to cause recession of the mercury meniscus and advancing the pump piston to return the meniscus to the reference mark, thereby determining the amount of mercury injected into the porous solid under various pressures, was repeated until the pressure of the nitrogen cylinder was reached.

A pressure-volume correction curve was established for the apparatus by carrying out a run as described above without a sample in the holder. The volume readings obtained when testing aggregates was corrected by subtracting amounts as

determined by this blank run at corresponding pressures.

<u>Calculations</u> The following method of calculation was taken from Ritter and Drake (38).

Let the total volume of all pores having radii between r and r + dr be

$$dV = D(r)dr 2$$

where D(r) is the distribution function for pore size. From Equation 1, assuming constant 6 and  $\Theta$ ,

$$pdr + rdp = 0$$
 3

Eliminating r and dr from Equations 1, 2 and 3 gives

$$dV = D(r) \frac{26 \cos \Theta}{p^2} dp = -D(r) \frac{r}{p} dp = 4$$

The volume measured by the mercury porosimeter is the volume of all pores having radii greater than r -- i.e., the total pore volume  $V_0$ , decreased by the volume V of pores smaller than r. Thus the pressuring curves plot  $V_0 - V$  as a function of p. The slope of the pressuring curve,  $\frac{d(V_0 - V)}{dp} = -\frac{dV}{dp}$ ,

is then an experimentally determinable quantity and equation 4 may be rewritten in the form:

$$D(r) = \frac{p}{r} \frac{d(V_0 - V)}{dp}$$
5

in which all the terms on the right are known or determinable. Values of the derivative in Equation 5 required to evaluate D(r)are readily obtained by graphical differentiation; r is calculated from Equation 1, and D(r) is calculated from Equation 5. Plotting D(r) against r gives the distribution curve.

It was arbitrarily decided to record the percentage of pores having radii equal to, or greater than 1 micron or 10,000 angstroms for purpose of comparison between the various aggregates. The total area under the pore-size distribution curve and the partial area under that curve between r = 0and r = 1 micron were measured with a planimeter and from these areas, the percentage of pores having radii equal to, or greater than one micron was determined. Throughout the calculations, the following constants were used:

Contact angle of mercury against the solid  $\Theta = 140^{\circ}$ Surface tension of mercury  $\delta = 480$  dynes per cm.

Instead of determining porosity and surface area of the aggregates separately, most laboratory methods use water, kerosene or oil absorption tests to design for the optimum binder content for a given mix.

In this study, the percent by weight of water absorption was obtained by the standard A.A.S.H.O-T 85-45 test. The absorption of SAE no. 10 oil by the coarse aggregates and of kerosene by the fine aggregates was determined by the Hveem's method of mix design (28).

To correlate the results of the routine laboratory tests and the actual absorption of asphalt by the aggregates under service conditions, pavement cores were collected from the four projects after different lengths of time under traffic, brought back to the laboratory and sawed in half to expose the inner surfaces.

The stone particles were then separated by visual inspection into absorptive and non-absorptive aggregates and subjected to X-ray diffraction analysis.

In another series of tests, the contour of each aggregate on the exposed surface of the core was traced with a Greist Micro-Scriber pencil.

The sample was then placed in an opaque projector and from its image projected on a screen, tracing was made of the external contour of the rock particle and the line of deepest asphalt penetration.

From the total area and the area imbibed with asphalt, measured by a planimeter, the percentage of asphalt absorption was calculated.

Statistical analyses were then made to determine:

1) the dependence of asphalt absorption on the source of the aggregates, e.g. on the projects;

2) the dependence of asphalt absorption on the size of the stone particles under examination; and

3) the dependence of asphalt absorption on the length of time the pavement has been exposed to traffic.

To ascertain the influence of absorption upon the hardening of the binder, the viscosities of the asphalt cements extracted from the pavement cores were determined.

Resistance to Stripping by Water

Most mineral aggregates attract water. What interests the engineer is whether a certain aggregate would prefer water to a given asphalt cement.

Most existing stripping tests use mixtures in a loose state and place heavy reliance on the judgment of an observer.

The A.S.T.M. Unconfined Compressive Strength test was chosen because of its quantitative character. It also measured the stability of the mixtures.

The materials used were mixes sampled from the batching plants and stored in closed cardboard boxes for two years in the laboratory. The molding and testing of the 4" x 4" specimens followed the Standard A.S.T.M. D 1074-54T and D 1075-54 methods.

Since the unconfined compressive strength depends on the internal friction and cohesion of the mix, sieve analyses were run on the extracted aggregates and softening point tests on the reclaimed asphalts, in order to give a correct interpretation of the results.

## RESULTS AND DISCUSSION

# Physical Properties of Bituminous Binders

The initial physical properties of the bituminous binders used are listed in Table 5.

Table 5. Initial physical properties of bituminous binders

Tests	Proj.	A Proj. B	Proj. C	Proj. D
Spec. grav. ASTM D 70-52	1.031	1.005	1.033	1.015
Soft. point, <sup>o</sup> F	114	115	116	114
Thin film res.(26)	127.0	128.5	125.0	126.0
Pen., 100g/5 sec. ASTM D 5-52 Thin film res. (26)	89 43	90 55	86 48	95 49
Thin film res.	4 <b>8.</b> 3	60.5	55.3	51.6
Loss on heat,% ASTM D 6-39T Thin film test (26)	0.08 0.30	0.07 0.09	0.07 0.08	0.08 0.11
Flash point, <sup>O</sup> F ASTM D 92-52	575	640	600	638
Sol. in CC14, % ASTM D 165-42	99.74	99.84	99.91	99.90
Ductility, cm ASTM D 113-44 Thin film res. (26)	150+ 145	150+ 79	150+ 150+	110+ 95
Spot test AASHO T 104-42	Neg.	Neg.	Neg.	Neg.
Visc., (11) 10 <sup>0</sup> poises	1.4	2.3	1.7	1.5
Asphaltene, % (6)	18.2	16.7	18.4	18.2
Sources of asphalts	Big Spring Texas	Eldorado Kansas	Sugar Creek Missouri	Norphdet Arkansas

٠.

#### Evaluation of tests

There is an apparent contradiction between the viscosity, penetration and softening point test results. According to the viscosity results, asphalt B was the hardest. However, based on the results of the tests run on the thin film residues, asphalt A seemed to harden the most, as shown by the loss on heat and penetration of thin film residues as a percentage of the original penetration.

Asphalt B registered the sharpest drop in ductility when thin film residues were tested. A study of the data suggests that all four asphalt cements were acceptable, and if there were any suspicion of danger from brittleness of the binders, it would center on asphalts A and B.

## Physical Characteristics of Aggregates

The sources and blending compositions of the aggregates used are given in Table 6.

Project	Material	Percent	Source
A 3	3/4-3/8" cr. stone	30	Ray Cook, NW $\frac{1}{4}$ 24-84-23, Story Co.
-3	3/8" Cr. stone	35	id.
Co	barse sand	25	Empire sand and gravel, NW $\frac{1}{4}$ 1-84-20 Marshall Co.
Fi	ine sand	10	SE $\frac{1}{4}$ 10-84-19, Marshall Co.

Table 6. Sources and blending compositions of aggregates

Table 6. (Continued)

Project	Material	Percent	Source	
В	3/4-3/8" cr. stone	35	Ft.Dodge Limestone NW $\frac{1}{4}$ 24-91-30, Humboldt Co.	
	-3/8" cr. stone	30	id	
	Pit run gravel	25	Ft.Dodge Limestone NE $\frac{1}{4}$ 34-90-30, Webster Co.	
	Fine sand	10	Davis Bros.,SW 🛓 1-89-29,Webster Co.	
С	3/4-3/8" cr. stone	32.5	Gendler NW 🛓 11-76-29,Madison Oo.	
	-3/8" cr. stone	37.5	id.	
	Coarse sand	20	Concrete Mtls. Co. West Des Moines	
	Fine sand	10	NE $\frac{1}{4}$ 24-77-36, Cass Co.	
D	3/4-3/8" cr. stone	30	Ray Cook, NW 🛓 24-84-23,Story Co.	
	-3/8" cr. stone	40	id.	
	Coarse sand	20	id.	
	Fine sand	10	SW $\frac{1}{4}$ 7-83-24, Story Co.	

## Mineral composition

Analysis: The X-ray diffraction patterns for the coarse aggregates from Projects A, B, C and D respectively are shown in Figures 3 to 6. The numbers appearing next to the name of the minerals denote the relative intensity of the peak one Figure 3. X-ray diffraction pattern of the coarse aggregates of Project A

.



Figure 4. X-ray diffraction pattern of the coarse aggregates of Project B



Figure 5. X-ray diffraction pattern of the coarse aggregates of Project C



Figure 6. X-ray diffraction pattern of the coarse aggregates of Project D



could expect at that particular angle of exposure.

Evaluation of tests: 1) The coarse aggregates of Projects A and D are rich in dolomite; those of Projects B and C, rich in calcite. 2) Dolomite is almost completely missing in the diffraction pattern of the coarse aggregates of Project B. These aggregates could therefore be classified as "limestones", if one were to use Pettijohn's nomenclature (35). 3) Even though it is strictly a qualitative test, an approximate calcite-dolomite ratio could be obtained from the graphs by measuring the relative heights of the calcite and dolomite peaks at their maximum intensity. The results would then be:

	Proj. A	Proj. B	Proj. C	Proj. D
% calcite	32.4	100.0	84.7	32.1
% dolomite	67.6	0	15.3	67.9

4. Using the same procedure but taking into account the presence of quartz, the results become:

	<u>Proj. A</u>	<u>Proj. B</u>	<u>Proj. C</u>	Proj. D
% calcite	27.8	87.1	75.9	29.6
% dolomite	58.0	0	13.7	62.6
% quartz	14.2	12.9	10.4	7.8

The results of the X-ray analysis are summarized in Table 7.

	Proj.	A	Proj. 1	Β	Pro	j. C	Proj	• D
	<u>Major M</u>	inors	<u>Major M</u>	inors	<u>Ma jor</u>	Minors	<u>Major</u> 1	linors
Coar	se					-		• .
agg	Dolo- ( mite d	Calcite quartz	Cal- Qu cite	uartz	Cal- cite	Dolo- mite quartz	Dolo- mite	Calcite quartz
Fine agg.	Quartz	Dolo- mite	Quartz	Cal- cite	Quartz	2	Quartz	Dolo- mite felds- par

Table 7. Mineral components of aggregates by X-ray

Determination of calcite and dolomite by EDTA The

results of this chemical analysis are given in Table 8.

Table 8. Determination of calcite-dolomite ratio

		Proj.A	Proj.B	Proj.C	<u>Proj</u>	)j.D	
					Reca	White	
%	calcite mean	43.95	99.18	82.33	24.61	31.77	
%	Dolomite mean	56.05	0.82	17.67	75.39	68.23	
S	tandard dev. for both	2.44	0.46	8.14	9.88	4.05	
No	. of deter- minations	5	5	9	7	9	

<sup>a</sup>Many of the coarse aggregates of Project D had a reddish color; the remainder, white. Tests were run separately on red and white particles of stone to determine the difference, if any, between them
The red-colored aggregates were also present in Project A, although in lesser numbers. The results given for Project A are for the composite samples, since no effort was made to separate the aggregates according to color in that case.

<u>Evaluation of tests</u> The following is the evaluation of tests:

1) The results confirm those given by the X-ray diffraction.

2) A reasonably good agreement exists between the chemical determination and the quantitative results obtained from the X-ray diffraction graphs in the manner indicated in part 1.

3) An analysis of variance gives an F ratio equal to 3.99, as compared to  $F_{0.05,1,14} = 4.60$ , taken from an Fdistribution table. Thus, the correlation between asphalt absorption and the reddish color of Project D aggregates cannot be proved.

4) Borrowing from Pettijohn's nomenclature (35), the following classification applies:

	<u>Proj. A</u>	<u>Proj. B</u>	Proj. C	Proj. D
Type of aggre- gates	Calcitic dolomites	Limestones	Dolomitic limestones	Calcitic dolomites

<u>Density</u> The bulk and powder specific gravities of the aggregates used are shown in Table 9.

Table 9. Bulk and powder specific gravities of aggregates

Property	Method	Proj. A	Proj. B	Proj. C	Proj. D
Bulk sp. gravity	AASHO T 85-45	CA=2.38	CA=2.54	CA=2.53	CA=2.33
Bulk sp. gravity	AASHO T 84-95	FA=2.58	FA=2.45	FA=2.57	FA=2.56
Powder sp gravity	•	CA=2.80 FA=2.70	CA=2.70 FA=2.65 LD=2.69	CA=2.69 FA=2.66 LD=2.69	CA=2.76 FA=2.70

In the table above, CA = coarse aggregate, passing the 3/4" and retained on the No. 4 sieves; FA = fine aggregate, passing the No. 4 and retained on the No. 200 sieves; LD = limestone dust, used only in Projects B and C to increase the proportions of aggregates passing the No. 200 sieve.

<u>Evaluation of tests</u> The results are in good agreement with the known fact that dolomite is a denser mineral than calcite.

<u>Texture and shape</u> The results are shown in Table 10. The terminology used was borrowed from Field (9) for shape and from Pettijohn (35) for texture.

<u>Evaluation of tests</u> Surface texture influences the adhesion between rock and bituminous material. Other things

	Proj. A	Proj. B	Proj. C	Proj. D
Texture	Dull, smooth (pits, stri- ations)	Glossy, rough (pits)	Glossy,rough (pits, ridges)	Dull, smooth (pits)
Shape	Angular (S.R.E.)*	crushed	crushed	angular (S.R.E.)*

Table 10. Texture and shape of the coarse aggregates

\*S.R.E. = slightly rounded edges

being equal, the binder would adhere better to aggregate B and C than A and D, because of the rough surface texture of the former. Aggregates B and C also have the edge as far as the stability of the mix is concerned, because of their shape.

Aggregate Resistance to Degradation

The degradation of the aggregates when they are used without binder is shown in Table 11.

## Evaluation of tests

1) The softest aggregates as measured by the Los Angeles Abrasion test turned out to have the lowest degradation ratio. This apparent contradiction must be due to the difference in the mechanisms by which the two tests try to break down the stone particles. The haphazard orientation of the coarse particles and the extent of the cushioning provided by the fines are other unknowns.

Sieve size	e G b	radation efore	Percent passing Gradations after compaction					
	С	ompaction	Proj.A	Proj.B	Proj.C	Proj.D		
3/411		100	100	100	100	100		
$1/2^{n}$		92	93	95	94	94		
3/8"		78	77	78	82	80		
No. 4	1	58	58	59	63	61		
No. 8	3	47	48	48	51	49		
No. 3	30	27	27	29	26	26		
No. 5	50	20	19	22	20	17		
No. 1	00	13	12	14	18	12		
No. 2	200	7	11	9	13	10		
Degra ratic	adat >	ion	1.53	1.32	2.10	1.53		
L.A. value	abr e	asion	32	34	28	32		

Table 11. Degradations of aggregates

2) At least there was excellent agreement between the two tests, as far as aggregates from Projects A and D are concerned.

<u>Aggregates and binders</u> Tables 12, 13, and 14 show the degradation of aggregates when combined with different amounts of 85-100 penetration asphalt cements and compacted by three different methods.

Evaluation of tests 1) The Double-Plunger Static compaction induced the greatest degradation of the aggregates. This might be attributed to the lack of freedom in this type of compaction for the coarse particles to reorient themselves

P1	roject	<u>A</u>				В	·		<u> </u>	
Sieve size	Original gradation		Grad	dation	after mix	xing, co	ompactin	g and to	esting	
3/4" 1/2" 3/8"	100.0 92.5 77.0	100.0 93.4 80.4	100.0 92.0 79.3	100.0 93.1 79.0	100.0 92.6 79.9	100.0 93.0 79.2	100.0 92.9 79.9	100.0 94.1 80.0	100.0 93.2 79.1	100.0 92.9 79.2
No. 4 No. 8 No. 30	57.5 46.0 26.5	60.4 48.4 28.7	60.5 48.1 28.9	59.3 47.8 29.2	61.2 48.6 28.6	60.3 48.5 27.9	59.8 48.2 27.6	60.8 48.3 29.2	60.4 48.0 29.4	60.7 48.7 29.8
No. 50 No. 100 No. 200	19.5 0 12.0 0 6.5	22.5 13.6	22.4 13.5 7.7	21.8 12.8 7.3	21.2 13.2 7.7	20.1 12.8 7.5	19.8 12.3 7.5	21.7 13.5 8.0	21.7 13.2 8.1	21.4 13.1 8.1
Asphalt L.A. va	t Content alue	4.3	5.6 32	6.8	4.6	5.2 34	5.9	4.6	5.6 28	6.0
Degradation ratio Filter-bitumen		1.29	1.28	1.17	1.29	1.24	1.22	1.33	1.34	1.3

Table 12. Degradation of aggregates due to Hveem's kneading compaction

P1	rojec	t		А			В		С		
Sieve size	e Or gr	iginal adation			Gradatic	on after	mixing	, compac	ting an	d testi	ng
3/4" 1/2" 3/8"	1	00.0 92.5 77.0	100.0 93.4 79.1	100.0 94.5 78.7	100.0 93.6 78.8	100.0 93.3 78.5	100.0 93.4 79.5	100.0 92.3 78.7	100.0 93.0 78.7	100.0 93.1 78.8	100.0 93.3 78.6
No. 4 No. 8 No. 3	4 3 30	57.5 46.0 26.5	59.7 47.6 27.9	59.4 47.4 27.8	59.0 47.3 28.3	58.7 47.1 27.9	59.0 47.5 28.2	58.5 46.9 28.1	59.9 47.6 27.0	59.3 46.9 27.3	58.9 46.7 27.6
No. 5 No. 1 No. 2	50 LOO 200	19.5 12.0 6.5	21.0 12.5 7.2	20.5 11.8 6.7	21.5 12.5 7.1	21.0 13.2 7.9	21.5 14.1 8.7	21.0 12.9 7.7	$     18.8 \\     12.1 \\     6.6   $	19.0 12.0 6.7	19.5 12.5 7.3
Aspha L.A.	alt c valu	ontent le	5.4	6.2 32	6.7	4.5	5.8 34	6.6	4.6	5.7 28	6.4
Degra	adati	on ratio	1.17	1.08	3 1.13	1.25	1.39	1.22	1.08	1.08	1.16
facto		. • • • • • • • • • •	1.10	1.03	1.03	1.21	1.34	1.19	1.02	1.04	1.12

Table 13. Degradation of aggregates due to Marshall's impact compaction

P	roject		A			B		C		
Sieve size	Original gradation			Gradatio	n after	mixing	, compa	cting ar	nd test:	ing
3/4" 1/2" 3/8"	100.0 92.5 77.0	100.0 94.8 84.2	100.0 94.4 83.7	100.0 95.9 84.6	100.0 95.3 83.7	100.0 94.3 84.3	100.0 95.1 85.4	100.0 95.3 85.5	100.0 95.9 84.8	100.0 95.4 85.6
No. 4 No. 8 No. 30	57.5 46.0 26.5	65.7 51.6 30.3	65.3 51.3 29.8	65.6 51.1 30.0	64.3 50.7 30.1	64.6 50.9 29.4	64.3 50.8 30.3	66.0 51.3 29.8	66.2 51.4 29.4	65.8 50.8 29.6
No. 50 No. 10 No. 20	$ \begin{array}{r}     19.5 \\     0 \\     0 \\     0 \\     6.5 \end{array} $	23.0 13.4 8.0	23.1 13.4 7.9	23.0 13.7 8.1	21.1 13.5 8.1	20.9 12.6 7.4	21.9 13.5 8.3	21.6 12.7 7.2	21.1 12.8 7.4	22.6 14.5 7.8
Asphal L.A. v	t Content alue	5.0	6 <b>.1</b> 32	7.1	4.2	5.3 34	6.2	4.3	5.4 28	6.4
Degrad	ation ratio	1.52	1.49	1.54	1.48	1.37	1.52	1.39	1.43	1.49
Filler factor	-bitumen	1.23	1.22	1.24	1.25	1.15	1.27	1.10	1.14	1.20

Table 14. Degradation of aggregates due to the A.S.T.M. Double-Plunger Static compaction

.

,

as compaction progresses.

2) Only the Marshall Drop-Hammer compaction gave results consistent with those obtained from the Los Angeles Abrasion test. This is not surprising, since both methods rely on impact to break the solid particles.

3) There seems to be no correlation between the bitumen content and the amount of degradation. Apparently the minute difference in film thickness was not enough to overcome the effect of the haphazard orientation of the aggregates in the mix.

4) The lubricating effect of the asphalt cements definitely reduced the aggregate degradation, except in the case of the static load compaction. Again, the explanation lies in the lack of movement of the solid particles, once leading has started.

<u>Road cores</u> In Table 15, the second line below the sieve sizes represents the job-mix recommended by the Iowa Highway Commission. The third line is the average gradation of the road cores, which have been subjected to traffic for one, two and three years, and which were located both under and between the traffic lanes.

<u>Evaluation of tests</u> There is no significant difference between the gradations of the first, second and third year cores.

3/4"	1/2"	3/8"	No.4	No.8	No.30	No.50	No.100	No.200	
	Project A								
100 100	98 91.1	78 80.2	63 62.1	50 48.6	30 28 <b>.5</b>	18 16.6	8.7 6.3	7.0 4.2	
	Project B								
100 100	90 92.3	76 80.9	59 60.3	50 48.1	29 28.8	19 18.4	11 8.9	6.1 4.8	
				Pro	ject C				
100 100	91 93.4	75 82.1	60 63.2	50 <b>49.</b> 9	30 28.9	19 17.1	8.6 6.4	5.6 3.8	
Project D									
100 100	88 92.0	75 81.7	60 63.4	50 51.1	28 28.6	19 17.6	11 9.0	7.9 5.7	

Table 15. Degradation of aggregates in road cores

The degradation ratios are all less than one; there are three possible conjectures to explain this puzzle.

1) The contractors did not use the required amounts of filler. This is very unlikely.

2) The extraction process was at fault. This hypothesis cannot stand, since the same procedure was used in part B.

3) Some of the fine aggregates have been incorporated into the asphaltic binder under the kneading action of traffic. If this were true, one would expect the asphalt cement to harden a great deal. This point cannot be cleared up without further intensive study. Absorptive Characteristics of the Aggregates

Total porosity and pore-size distribution of the coarse aggregates are shown in Table 16.

	Proj.A	Proj.B	Proj.C	Proj.D	<u></u>
Total poro- sity, %	15.0	6.0	6.0	15.6	
Pores having radii greater than, or equal to 1 micron, as a percentage of all pores	19.4	10.2	6.7	29.1	

Table 16. Total porosity and pore-size distribution

The pore-size distribution curves are shown in Figures 7 to 10.

The evaluation of test is as follows:

1. The dolomitic rocks of Projects A and D are more porous than the calcitic rocks of Projects B and C, even though they are denser than the latter.

2. The pores of the dolomitic rocks are also larger than those of the calcitic rocks.

3. The asphalt cements in Projects A and D are therefore expected to harden faster than those in Projects B and C, other conditions being equal.

The results of water, oil and kerosene absorption by the aggregates are given in Table 17.

Figure 7. Graph of the size distribution function of the coarse aggregates of Project A



Figure 8. Graph of the size distribution function of the coarse aggregates of Project B



Figure 9. Graph of the size-distribution function of the coarse aggregates of Project C





Figure 10. Graph of the distribution function of the coarse aggregates of Project D

.



Wat Project % 1	ter abs. by wt.	SAE No. 10 oil abs., % by wt.	Kerosene absorp- tion, % by wt.
A	······		
Coarse aggrega	ate 6.73	5.60	
Fine aggregate	e 1.67		2.30
В			
Coarse aggrega	ate 2.10	3.50	
Fine aggregate	e 2.46		4.60
С			
Coarse aggrega	ate 2.26	3.80	
Fine aggregate	e 1.34		3.20
D			
Coarse aggrega	ate 7.21	5.60	
Fine aggregate	2.00		2.20

Table 17. Water, oil and kerosene absorption

Evaluation of tests is as follows:

1. The water and oil absorptions are consistent with the porosity characteristics of the aggregates.

2. The porous aggregates absorb more water than oil. The reverse is true with the less porous limestones. In the latter case, since the pores are fewer and smaller, the surface energy relationships probably assume more importance.

X-ray diffraction analysis: The X-ray diffraction patterns of the non-absorptive and absorptive are shown in Figures 11 to 16.

Evaluation of tests is as follows:

An examination of Figures 11, 12 and 15, 16 shows that the difference between very slight and appreciable absorption

Figure 11. X-ray diffraction pattern of the coarse aggregates of Project A with appreciable asphalt absorption

مر.





Figure 12. X-ray diffraction pattern of the coarse aggregates of Project B with negligible asphalt absorption

/



Figure 13. X-ray diffraction pattern of the coarse aggregates of Project C with no asphalt absorption

.



Figure 14. X-ray diffraction pattern of the coarse aggregates of Project C with some asphalt absorption

,



Figure 15. X-ray diffraction pattern of the coarse aggregates of Project D with very slight asphalt absorption

•



Figure 16. X-ray diffraction pattern of the coarse aggregates of Project D with a great deal of asphalt absorption

•



lies in the calcite-dolomite ratio. The greater the percentage of dolomite, the greater the absorption. This agrees with the previous finding that the dolomitic aggregates are more porous than the calcitic. Figures 12 and 13 show the diffraction patterns of two non-absorptive aggregates, where the dolomite peaks are practically absent.

Quantitative determination of absorption: The statistical analyses in this section were made according to the methods presented by Snedecor (40).

The relationship between asphalt absorption by the aggregates and the various projects is shown by the following data and analysis:

<u>Project</u>	Sum ZX <sub>ii</sub> *	No. of determ.	Mean	
	-5	ni	x	
А	1673	62	27.0	
В	1174	55	21.3	
С	1130	62	18.2	
D	1895	59	32.1	

 $X_{ij}$  = the individual determination of the percent asphalt absorption; i stands for the project and j for the determination.

To test the null hypothesis  $H_0 : K_A^2 = 0$ , e.g., the absorption of asphalt by aggregates is not dependent upon the projects, the following analysis was made:

Source of variation	<u>d.f.</u>	<u>* S.S.*</u>	M.S.*	E.M.S.*
Projects	3	6,789	2,263	$\sigma^2 + n_0 \kappa_A^2$
Determinations	234	73,732	315	$\sigma^2$
Total	237	80,520		

D.f. = degrees of freedom, S.S. = sum of squares, M.S. = mean squares and E.M.S. = expected mean square.

$$n_{0} = \frac{1}{a-1} (n - \frac{n_{1}}{n}) = \frac{1}{4-1} (238 - \frac{14,194}{238}) = 59.3$$

$$s^{2} = 315 \text{ estimates } \sigma^{2};$$

$$s_{A}^{2} = (2,263 - 315)/59.3 = 32.8 \text{ estimates } K_{A}^{2}.$$

$$\underline{\text{Test}}: F = \frac{2,263}{315} = 7.18 \quad \text{Cf. } F_{0.05,3,234} = 2.64$$

The hypothesis  $H_0$  is rejected, because the F value exceeds that given in the F-distribution table for a 95% confidence interval and the degrees of freedom  $f_1 = 3$  and  $f_2 = 234$ .

Thus the amount of asphalt absorption varies according to the projects.

Since the coarse aggregates of Projectsa A and D came from the same source, it might be instructive to make a statistical test on these two projects alone.

Source of variation	<u>d.f.</u>	<u>S.S.</u>	<u>M.S.</u>	<u>E.M.D.</u>
Projects (A and D)	1	798	798	$\sigma^2 + n_0 K_A^2$
Determinations	119	47,899	403	$\sigma^2$
Total	120	48,697		

 $\begin{array}{l} n_{0} = 60.5 \\ s^{2} = 403 \ \text{estimates } \sigma^{2}; \\ s_{A}^{2} = (798 - 403)/60.5 = 6.5 \ \text{estimates } \mathbb{K}_{A}^{2} \ . \\ \hline \underline{\text{Test:}} \quad F = \frac{798}{403} = 1.98 \quad \text{Cf. F}_{0.05,1,119} = 3.92 \\ \hline \text{Since } 1.98 \quad 3.92, \ \text{the hypothesis } H_{0} \ \text{is accepted; e.g., there} \\ \hline \text{is } \underline{no} \ \text{significant difference between the aggregates of Projects A and D, as far as asphalt absorption is concerned.} \end{array}$ 

The next step was to classify the projects according to the absorptive capacity of their aggregates. This was done by means of Hartley's Sequential Method of comparing the means (40).

The test consists in computing a series of differences  $D_i$ , which are significant at the 5% level, then comparing them with the a(a-1)/2 sample differences in the experiment.

 $D_{i}$  are the products of  $s_{\overline{X}}$  and a factor  $Q_{i}$  taken from a table (40).

In this case,  $s_{\overline{x}} = 315/59.3 = 2.31$ 

<u>Project</u>	<u>x</u>	<u>x - 18.2</u>	<u>x</u> - 21.3	<u>x</u> - 27.0
D	32.1	*13.9 (8.4)	*10.8 (7.7)	5.1 (6.4)
А	27.0	* 8.8 (7.7)	5.7 (6.4)	
В	21.3	3.1 (6.4)		
С	18.2			

The \* indicates significant differences.

Evaluation of tests: The following differences between means are significant.

- a) Project D and Project C
- b) Project A and Project C
- c) Project D and Project B.

However, the cross-sectional areas of the aggregates examined might have influenced the amount of absorption. A statistical analysis of aggregate sizes versus projects would answer the question

Source of variation	<u>d.f</u> .	<u>S.S.</u>	<u>M.S.</u>	E-M-S-
Total	398	12.186413		
Project	3	0.272978	0.09099	$\sigma^2 + n_0 \kappa_A^2$
Sizes	395	11.913435	0.03012	σ <sup>2</sup>

<u>Test</u>:  $F = \frac{0.09099}{0.03016} = 3.02$  Cf.  $F_{0.05,3,395} = 2.62$ 

Since 3.02 2.62, the null hypothesis  $H_0 : K_A^2 = 0$ , e.g., the sizes of the aggregates in the four projects which were examined for absorption were the same, is rejected.

Evaluation of test: Thus the quantitative study of absorption was not conclusive. This might be due to the fact that all the pavement cores were lumped together, without regard to their length of service.

A final test was made in which the pavement cores were separated according to project and length of time under traffic. Then, for each project, an analysis of covariance was run to find the difference, if any, between the absorption after two years and three years of service (referred to as second and third coring, respectively). The covariants are x = size of the aggregate particle in square inches and y =percent absorption.

The analysis of Project A is presented in the Appendix as a sample of the calculations. No test was made on Project D since the pavement in question has been in service only for two years.

Evaluation of tests: For both Projects A and C, it was found that there was no difference in asphalt absorption between the second and third coring.

However, in the case of Project C, it was found that the slopes of the two regressions were not equal. Since the slope of the regression line for the third coring is flatter than that for the second coring, and both are negative, this means that the small particles had absorbed about all the asphalt they could, whereas the coarse aggregates were still absorbing asphalt up to the time of the third coring.

In all cases, the slopes of the regression are negative. This means that the small particles absorb proportionately more asphalt than the larger ones.
Viscosity changes in the road cores: The changes in the viscosity of the asphalt cements in the pavements under the combined action of traffic and weathering are shown in Tables 18 and 19.

Time under traffic in months	Proj. A	Proj. B	Proj. C	Proj. D	
0 10	1.4 7.6	2.3 3.9	1.7 2.9	1.5	
16 25 33	8.6	6.5	3.2	6.5	

Table 18. Viscosities in 10<sup>6</sup> poises

Table 19. Percent of viscosity increase after about one year in service

	Proj. A	Proj. B	Proj. C	Proj. D
Percent in- crease in viscosity	443	70	71	333

Evaluation of tests: These results confirm the suspicion inspired by the knowledge of the porosity of the four aggregates. The extreme hardening of the asphalt cements of Projects A and D could be caused by any one of the following two phenomena: 1) Selective absorption leaves the asphalt film richer in asphaltenes; or 2) Absorption of asphalt into the pores of the aggregates depletes the binder content and makes the films thinner, thus more susceptible to oxidation.

### Resistance to Stripping by Water

The indexes of retained strength for the four projects are given below:

# Proj. A Proj. B Proj. C Proj. D

Index of retained strength 88.1% 91.2% 64.4% 76.6% Project C material came out the worst. However, since there is as yet no general agreement as to what minimum value of retained strength would constitute the danger point, it cannot be said categorically that failure will occur on Project C due to stripping. Besides, this could not be blamed on the aggregates alone. Project C aggregates might give good adhesion with another asphalt cement.

On the basis of this test alone, pavements C and D must however be watched carefully for failure due to water action.

Other interesting data are the percentages of change in height based on the original height in the unconfined compression test:

	F -	roj. A	Proj. B	Proj. C	Proj.D
Average % deflection	Dry	2.45	1.65	2.65	1.40
Sc	aked	3.03	2.49	3.49	3.39
% decrease in height from soaking		23.7	50.9	31.7	142.1

There does not seem to be any correlation between these

deflections and the indexes of retained strength.

However the index of retained strength presents only half the picture. A mix might have a very high index of retained strength, but if its dry uncompressive strength is very low to start with, it would not be much good. The maximum and minimum unconfined compressive strengths, both dry and soaked are given below.

	Unc	onfined	compr	essive s	trengt	hs in ps	i	
		Proj.A	P	roj. B	Pr	oj.C	Pro	j. D
	Dry	Soaked	Dry	Soaked	Dry	Soaked	Dry	Soaked
Maximum	786	691	858	842	<b>1</b> 049	643	500	<b>41</b> 4
Minimum	738	635	83 <b>1</b>	722	898	571	498	406

These data show that, even though Project C mix has the lowest index of retained strength, it has the highest dry unconfined compressive strength. On the other hand, Project D mix, which has the second lowest index, also has the lowest dry unconfined compressive strength. In the preceding paragraph, it was also found that Project D mix gave the greatest decrease in height due to soaking. Thus, Project D gives some indication as being the most critical.

The study would not have been complete without a determination of the consistency of the asphalt cements, the binder contents and the gradation of the aggregates in those mixes. The changes in softening points of the asphaltic binders are shown below: (the softening points are given in degrees Fahrenheit)

	Proj. A	Proj. B	Proj.C	Proj. D
Origina1	114	115	116	114
Final	135	145	126	131
Percent change	+18.4	+26.1	+8.6	+14.9

Project B asphalt was found to have hardened the most. Since all the mixes have been kept in closed cardboard boxes, the hardening was probably caused by volatilization. Apparently, the rigidity of the bitumen rendered displacement by water more difficult under these conditions.

Asphalt C has changed surprisingly little. The low index of retained strength must therefore be due to the relatively low increase in softening point and high ductility.

The data on aggregate gradation and binder content are shown in Table 20. These figures represent the averages of four determinations.

The gradation of the different specimens within a given mix was surprisingly uniform. The same could be said of the binder content. This might be attributed to the good field sampling and the quartering done in the laboratory. However, it seems more likely that the large size of the samples tend to minimize the sampling variations.

Sieve	Percent passing							
size —	Proj. A	Proj. B	Proj. C	Proj. D				
3/4"	100.0	100.0	100.0	100.0				
1/2"	93.7	95.5	95.3	95.5				
3/8"	85.1	87.2	88.4	87.9				
No. 4	65.8	66.8	72.7	70.3				
No. 8	50.7	52.2	56.5	57.3				
No. 30	31.9	31.2	32.0	34.0				
No. 50	21.0	21.9	20.4	23.1				
No. 100	10.1	11.6	9.5	12.8				
No. 200	6.8	7.0	6.6	7.9				
% asph. by wt. of aggregates	6.8	6.6	6.6	6.7				

Table 20. Aggregate gradations and binder contents

As far as variables in the unconfined compressive strength determination, gradation and binder content could therefore be ruled out.

#### CONCLUSION

This study was limited to the aggregates retained on the 3/8" sieve. There is no doubt that the fine aggregates and the filler material would influence the durability of bituminous mixtures.

But even within that limited scope, many important phases of the problem had to be neglected. Thus no properties associated with the surfaces of the aggregates, such as surface area, coatings, surface tension, chemical reactivity, etc. were investigated. This does not mean however, that they are of less importance.

It must be emphasized that the findings summarized below are applicable only to the materials and the traffic and weather conditions stated at the beginning of the paper. No generalization is therefore warranted.

The investigation discloses that:

1) The coarse aggregates of Projects A and D are richer in dolomite and have greater specific gravity than those of Projects B and C. Although all four aggregates are crushed rocks, those of Projects B and C have sharper edges and rougher texture. Logically this should favor the adhesion of asphalt to hose aggregates, with adequate mixing. However, a microscopic study of the grain-size characteristics of the aggregates would probably shed more light on their adhesive

properties than the calcite-dolomite ratio.

2) The dolomitic rocks have more and larger pores than the calcitic rocks. There are indications that asphalt absorption and asphalt hardening are closely related to the porosity and pre-size distribution of the coarse aggregates. The greater the porosity and the larger the pore openings, the greater the absorption and the faster the rate of asphalt hardening.

3) The Marshall Drop-Hammer compaction degrades the aggregates in the same manner as the Los Angeles Abrasion test. The presence of fines and binder minimizes the degradation, except in the case of the Double-Plunger Static compaction where the aggregates are not free to reorient themselves. The asphalt content does not appear to be a variable in these degradation processes, at least in the amounts normally used.

4) No comparison with traffic degradation is possible, because the amount of filler in almost every pavement core taken was less than that required by the job-mix formula. This suggests the idea that some of the fine aggregates had been incorporated into the binder. However, this hypothesis cannot be proved without further study.

5) All four mixes show good resistance to the stripping action of water. However, the mixes from Projects C and D seem more vulnerable than the other two mixes. Softening point results indicate that asphalt B has hardened the most. This probably explains its high index of retained strength,

since the rigidity of the binder renders the displacement of the asphalt film by water more difficult. This change appears to come under the classification of steric hardening (4). In this respect, it might be profitable to determine the amount of insoluble residues in hydrochloric acid, because if they were clay minerals, they could exert more influence on adhesion than calcite or dolomite.

6) As far as the mineral composition of limestone is concerned, X-ray diffraction and chemical analysis using EDTA give very similar results. It seems possible to obtain approximate quantitative results from the X-ray diffraction patterns by measuring the heights of the peaks of the various minerals at their greatest intensity.

The overall conclusion of this investigation is that the porosity and pore-size distribution of the aggregates are worthwhile items to study, because of their great influence on the absorption and, indirectly, on the hardening of the asphalt cement.

## LITERATURE CITED

- American Association of State Highway Officials. Standard specifications for highway materials and methods of sampling and testing. Part 2. 6th ed. The Association. Washington, D. C. 1950.
- Bisque, R. E. Analysis of carbonate rocks for calcium, magnesium, iron and aluminum with EDTA. (Typewritten) Colorado School of Mines. Golden, Colorado. 1959.
- Blanks, R. F. Modern concepts applied to concrete aggregates. Proc. Amer. Soc. Civil Engrs. 75:441-468. 1949.
- 4. Brown, A. B., Sparks, J. W. and Smith, F. M. Steric hardening of asphalts. Proc. Assoc. Asphalt Paving Technologists. 26:486-494. 1957.
- 5. Charles, R. J. Energy-size reduction relationships in comminution. Mining Engineering. 9:80-88. 1957.
- Csanyi, L. H. and Fung, H. P. A rapid means of determining the constituents of asphalts. Proc. Assoc. Asphalt Paving Technologists. 23:64-77. 1954.
- Donaldson, J. A., Loomis, R. J. and Krchma, L. C. The measurement of aggregate absorption. Proc. Assoc. Asphalt Paving Technologists. 16:353-372. 1947.
- 8. Ebberts, A. R. Adhesion tension measurements. Proc. Assoc. Asphalt Paving Technologists. 8:68-77. 1937.
- Field, F. The importance of percent crushed in coarse aggregates as applied to bituminous pavements. Proc. Assoc. Asphalt Paving Technologists. 27:294-322. 1958.
- Goetz, W. H. Methods of testing for water resistance of bituminous paving mixtures. Amer. Soc. Testing Materials. Special Technical Publication 240:84-94. 1958.
- Griffin, R. L., Miles, T. K. and Penther, C. J. Microfilm durability test for asphalts. Proc. Assoc. Asphalt Paving Technologists. 24:31-62. 1955.

- 12. Grim, R. E. Clay mineralogy. McGraw Hill Book Company, Inc. New York, N. Y. 1953.
- Gzemski, F. C. Factors affecting adhesion of asphalt to stone. Proc. Assoc. Asphalt Paving Technologists. 17:74-92. 1948.
- 14. Hassler, G. L., Brunner, E. and Deahl, T. J. The role of capillarity in oil production. Trans. Amer. Inst. of Mining and Metallurgical Engrs. 155:155-174. 1944.
- 15. Holmes, J. A. A contribution to the study of comminution A modidied form of Kick's law. Trans. Institution of Chemical Engrs. 35:125-156. London. 1957.
- Hubbard, P. Adhesion of asphalt to aggregates in the presence of water. Proc. Highway Research Board. 18, part 1:238-249. 1938.
- Huber, C. F. and Thompson, P. F. The function and chemistry of asphalt additives. Proc. Assoc. Asphalt Paving Technologists. 24:374-391. 1955.
- Hveem, F. N. Use of the centrifuge kerosene equivalent as applied to determine the required oil content for dense graded bituminous mixtures. Proc. Assoc. Asphalt Paving Technologists. 13:9-40. 1942.
- 19. Iowa Geological Survey. First and second annual report of progress by the state geologist and the assistant and chemist on the geological survey of the State of Iowa. [Iowa Geological Survey] Des Moines, Iowa. 1867-1868.
- 20. Iowa Geological Survey. Sketch of the geology of Iowa. [Iowa Geological Survey] Des Moines, Iowa. 1920.
- 21. Iowa State Highway Commission. Standard specifications for construction on primary, farm to market and secondary roads and maintenance work on the primary road system. [Iowa State Highway Commission] Ames, Iowa. 1960.
- 22. Knight, B. H. Influence of aggregate type in stability of asphalt mixtures. Jour. Appl. Chemistry. 3: 337-344. 1953.

- 23. Knight, B. H. and Knight, R. G. Road aggregates and their uses and testing. Edward Arnold and Co. London, England. 1948.
- 24. Lettier, J. A., Fink, D. F., Wilson, N. B. and Farley, F. F. Mechanism of absorption of bituminous materials by aggregates. Proc. Assoc. Asphalt Paving Technologists. 18:278-300. 1949.
- 25. Leverett, M. C. Capillary behavior in porous solids. Trans. Amer. Inst. of Mining and Metallurgical Engrs. 142:152-169. 1941.
- 26. Lewis, R. H. and Welborn, J. Y. The properties of the residue of 50-60 and 85-100 penetration asphalts from oven tests and exposures. Public Roads. 22:27-48. 1941.
- Melville, P. L. Weathering study of some aggregates. Proc. [U. S.] Highway Research Board. 28:238-248. 1948.
- 28. Mix design methods for hot mix asphalt paving. 1st ed. The Asphalt Institute. Asphalt Institute Building, University of Maryland. College Park, Maryland. 1956.
- Nellensteyn, F. J. Theoretical aspects of asphaltic bitumen in the light of recent research. Proc. Assoc. Asphalt Paving Technologists. 8:78-84. 1937.
- 30. Nevitt, H. G. and Krchma, L. C. Absorption of liquid bituminous cements by aggregates. Proc. Assoc. Asphalt Paving Technologists. 13:52-68. 1942.
- 31. Parr, W. K. Field observations of the behavior of bituminous pavements as influenced by moisture. Amer. Soc. Testing Materials. Special Technical Publication 240:3-16. 1958.
- 32. Patton, J. and Reeder, W. New indicator for titration of calcium with (ethylenedinitro) tetraacetate. Anal. Chemistry. 28:1026-1028. 1956.
- 33. Pauls, J. T. and Goode, J. F. Application and present status of the immersion-compression test. Proc. Assoc. Paving Technologists. 16:373-393. 1947.
- 34. Pauls, J. T. and Rex, H. M. A test for determining the effect of water on bituminous mixtures. Public Roads. 24:115-129. 1945.

- 35. Pettijohn, F. J. Sedimentary rocks. Harper and Bros. New York, N. Y. 1949.
- 36. Purcell, W. R. Capillary pressures Their measurement using mercury and the calculation of permeability therefrom. Trans. Amer. Inst. of Mining and Metallurgical Engrs. 186:39-48. 1949.
- 37. Rice, J. M. Relationship of aggregate characteristics to the effect of water on bituminous paving mixtures. Amer. Soc. Testing Materials. Special Technical Publication 240:17-34. 1958.
- Ritter, H. L. and Drake, L. C. Pore-size distribution in porous materials. Ind. Eng. Chem. Anal. ed. 17: 782-786. 1945.
- 39. Rush, E. F. Petrography and physical properties of some devonian limestones of Iowa. Unpublished M.S. Thesis. Library, Iowa State University of Science and Technology, Ames, Iowa. 1957.
- 40. Snedecor, G. W. Statistical methods. 5th ed. The Iowa State College Press. Ames, Iowa. 1956.
- 41. Spielmann, P. E. and Hughes, A. C. Asphalt roads. Edward Arnold and Co. London, England. 1936.
- 42. Stross, F. H. and Anderson, A. P. Stripping versus coating of mineral aggregates. Proc. Assoc. Asphalt Paving Technologists. 13:1-8. 1942.
- Thelen, E. Surface energy and adhesion properties in asphalt-aggregate systems. [U.S.] Highway Research Board Bul. 192. 63-74. 1958.
- 44. Turner, R. S. and Wilson, J. D. Degradation study of some Washington aggregates. State College of Washington. Washington State Institute of Technology Bul. 232. 1956.
- 45. Tyler, O. R. Adhesion of bituminous films to aggregates. Purdue University. Eng. Exp. Sta. Bul. 62. 1938.
- 46. U. S. Weather Bureau. Climatological data Iowa. Vol. 70. 1959.

- 47. Weissmann, R. C. and Diehl, H. A new method utilizing versene for the determination of the calcite-dolomite ratio in carbonate rocks. Proc. Iowa Acad. Sci. 60: 433-437. 1953.
- 48. Winterkorn, H. F. Surface chemical aspects of the bond formation between bituminous materials and mineral surfaces. Proc. Assoc. Asphalt Paving Technologists. 7:79-85. 1936.

#### ACKNOWLEDGMENT

The writer wishes to express his sincere appreciation to L. H. Csanyi, Professor of Civil Engineering, in charge of the Bituminous Research Laboratory at Iowa State University, for his valuable suggestions, criticism and encouragement.

Thanks are also due to Dr. H. P. Fung, Associate Professor of Civil Engineering at Iowa State University, in charge of Project 346-S, for his kind guidance and help and to the Iowa Highway Research Board for sponsoring the Project.

The writer wishes to express his gratitude to Dr. R. L. Handy of the Iowa Engineering Experiment Station for his permission to use the X-ray Defraction Unit and to Dr. J. Lemish of the Geology Department for making available to him the mercury-pressure porosimeter.

The author is also indebted to his many co-workers in the Bituminous Research Laboratory, whose cheerful cooperation is hereby gratefully acknowledged.

APPENDIX

-

							- 157		
$\Sigma x^2$ C $\Sigma x^2$		1.76 <u>1.08</u> 0.68	8548 2727 5821	ΣΧΥ C Σ×y	= 94 = <u>118</u> = -23	.6467 .1327 .4860	$\sum_{C} \frac{y^2}{z^2} = \frac{z^2}{z^2} = \frac{z^2}{z$	15,608.89 <u>12,888.87</u> 2,720.02	
Tab:	le	22.	Projec (n = 2	t A, 8)	third	coring	;	0.225, $\overline{y} = 29.2$	
$\Sigma x^2$	=	2.13 1.41	8102 6600	ΣXY C	= 135 = <u>184</u>	.4808 .0141	$\Sigma Y^2 = C =$	33,790.01 23,903.13	
Σx <sup>2</sup>	=	0.72	1502	∑xy	= -48.	• 5333	$\Sigma y^2 =$	9,886.88	
			Draica	+ .	+ - + - 1		(n -	42)	

Table 21. Project A, second coring;  $\overline{x} = 0.269$ ,  $\overline{y} = 29.3$ (n = no. determinations = 15)

Table 23. Project A, total coring,	(n	=	43)	
------------------------------------	----	---	-----	--

ΣΧ	=	10.328	<b>x</b> <sub>T</sub> =0.240	$\Sigma x^2 = 3.906650$	ΣXY=230.1275	xy <sup>2</sup> = 49,398.90
ΣΥ	=	1257.8	<del>y</del> T=29.3	C = 2.480641 $\Sigma x^{2} = 1.426009$	C= <u>302.1060</u> Σxy=-71.9785	$C = \frac{36,792.10}{2y^2 = 12,606.80}$

The purpose of the test was to examine the three following hypotheses:

- 1. The 2 samples were drawn from normal populations with common  $\sigma^2$ ; e.g.,  $H_0 : \sigma_1^2 = \sigma_2^2$ .
- 2. The slopes of the 2 regressions are the same; e.g.,  $H_0 : B_1 = B_2$ .

3. The 2 regressions coincide; e.g.,  $H_0 : a_1 = a_2$ . This hypothesis needs not be taken up, unless the first two were accepted.

The first question was answered by Bartlett's of homogeneity of variance, whose computations are shown below:

Table 24. Computations of homogeneity of variance

Samı	p1e	∑x <sup>2</sup>	d.f. n-1	Reciproc 1/(n-1)	al M.S. s <sup>2</sup>	log s <sup>2</sup>	(n-1)log s <sup>2</sup>
1	6,8	97.80	14	0.0714	492.70	2.69258	37.69612
2	9,8	86.89	27	0.0370	366.18	2.56370	69.21990
a=2	16,7	84.69	41	0.1084			106.91602

 $\overline{s}^{2} = \sum x^{2} / \Sigma(n-1) = \frac{16,784.69}{41} = 409.383$   $(\log \overline{s}^{2}) \Sigma(n-1) = (2.61213)(41) = 107.097$   $x^{2} = (2.3026) [(\log \overline{s}^{2}) \Sigma(n-1) - \Sigma(n-1)(\log s^{2})]$  = 0.42.

Since  $x_{0.05,1}^2 = 3.84$ , the hypothesis is accepted. The two variances are the same.

The other two questions are answered in the Table 25.

						De	viati	ons from regre	ssion
Line	Coring	f	∑x <sup>2</sup>	Σχγ	Σy <sup>2</sup>	Reg. coef.	f	$\sum_{(\Sigma y)^2/\Sigma x^2}$	Mean square
1 2	Second Third	14 <u>27</u>	0.685822 0.721502	-23.4860 - <u>48.5332</u>	2,720.0 9,886.8	$\frac{12}{9}$ -34.2	13 <u>26</u>	1,915.74 6,622.22	147.36 254.70
3 4 5 6 7	Within Reg.coe Common Adj.mea Total	f. 41 1s 42	1.407324 1.426010	-72.0192 -71.9790	12,606.9 12,606.8	91 -51.2 80	39 1 40 1 41	8,537.96 383.40 8,921.36 52.20 8,973.66	218.92 383.40 223.03 52.20

Table 25. Analysis of covariance of asphalt absorption versus time under traffic for Project A

 $H_0: B_1 = B_2$  (B stands for the slope of the regression line).

Test: 
$$F = \frac{M.S. \text{ for regression coefficients}}{M.S. \text{ within samples}} = \frac{383.40}{218.92}$$

= 
$$1.75$$
 Cf.  $F_{0.05,1,39} = 4.09$ .

Since 1.75 < 4.09, the hypothesis  $H_0$  is accepted; e.g., the 2 regression lines are parallel.

$$H_0 : a_1 = a_2$$
 [a stands for the mean  $(\overline{x}, \overline{y})$ ]

Test:  

$$F = \frac{M.S. \text{ for adjusted means}}{M.S. \text{ for common reg.}} = \frac{52.20}{223.03}$$
 1

Since F is less than 1, the model or the way the test was set up might be wrong. To find out, the inverse of F or F' was tested:

$$F' = \frac{223.03}{52.20} = 4.2$$
 Cf.  $F_{0.05,40,1} = 251$ . Since

4.2 < 251, ordinary sampling variations were to be blamed. The final conclusion is that the 2 populations have the same regression lines.

.

•