Commonwealth of Kentucky Department of Highways

# A LABORATORY INVESTIGATION OF MINERALOGICAL, CHEMICAL AND PHYSICAL PROPERTIES OF LIMESTONE AGGREGATES

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### INTRODUCTION

The project described in this report represents one phase of comprenhensive research with all types of aggregates for highway paving mixtures in Kentucky. It was designed to show the fundamental properties of limestones which determine their suitability for this type of use --- particularly their so-called durability. For this reason, emphasis was placed on analysis of the intrinsic properties of the aggregates themselves rather than analysis of procedures or test methods, such as freezing and thawing.

Ultimately, of course, such tests must be performed in order to accomplish the differentiation, and some tests of this type have been included to date. However, no effort has been made to evaluate these procedures. Inasmuch as only a part of the work originally outlined has been completed, this necessarily serves as a report of progress to be supplemented from time-totime in the future.

#### PROCEDURE

In order to isolate limestone aggregate showing distinct ranges in performance, four quarries were initially chosen and generally rated as to their influence on concrete pavements. This rating was made from performance surveys of pavements in service. \*Slide No. 1 illustrates a heavily traveled pavement, (U.S. 25) made with aggregate from Quarry No. 2, which was rated as good. This pavement has 21 years of service.

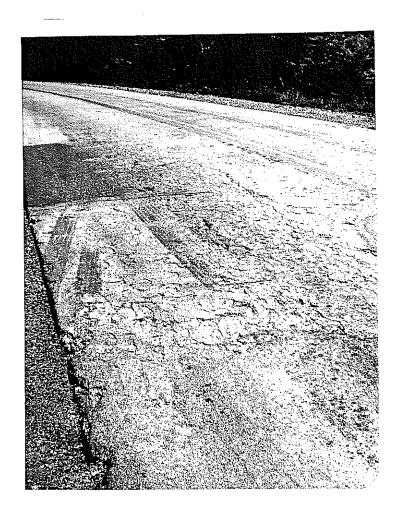


Slide No. 1. U.S. 25 - Berea-(Roundstone)

Slide No. 2 shows a failing pavement on U.S. 60, which is less heavily traveled than U.S. 25 and which has 24 years of service. This concrete was made with aggregate from Quarry No. 3, which is rated as poor. Of the two remaining sources, Quarry No. 1 was rated fair on the basis of extensive service records and Quarry No. 4 as excellent on the basis of more restricted use and pavements of more recent origin. The sources

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rated as fair and poor have been abandoned for commercial use. Early in the laboratory investigation, it became apparent that quarries as a whole and general service records were not discriminating enough to definitely evaluate the intrinsic properties of the limestone, as they varied throughout the quarry. For example, when the pavement shown in Slide No. 2 was inspected closely (Slide No. 3) almost every failure could be traced to one distinctive type of aggregate. This represented a ledge less than six feet in thickness in the quarry.



Slide No. 2. U.S. 60 - Made from Quarry No. 3

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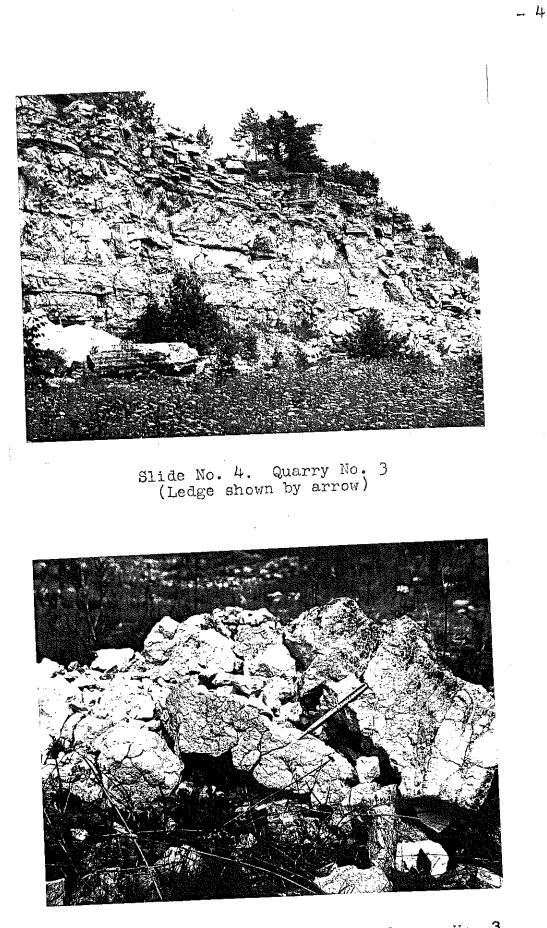


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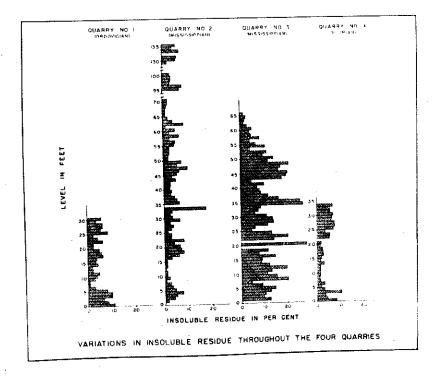
Slide No. 3. U.S. 60 - Made from Quarry No. 3 Examination of Quarry No. 3 (Slide No. 4) showed this ledge to be a pure cream colored, fine-grained limestone, which had the appearance of being quite sound. However, blocks of this material on the quarry floor had disintegrated almost completely in comparison to other materials (Slide No. 5).

The quarries were analysed thoroughly on a foot by foot basis. Samples were obtained at one foot vertical intervals from the quarry floor to the point where stripping operations commence.

The limestone was first described as it appeared under the microscope. It was digested in 6N Hydrochloric Acid and the amount and type of insoluble residue was described. Next, thin sections of each foot were made from a vertical and horizontal plane with intent to define structure. The Bulk Specific Gravity and Absolute Specific Gravity were determined



Slide No. 5. Weathered Block in Quarry No. 3



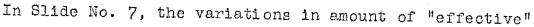
Slide No. 6. Variations in Insoluble Residue Throughout the Four Quarries.

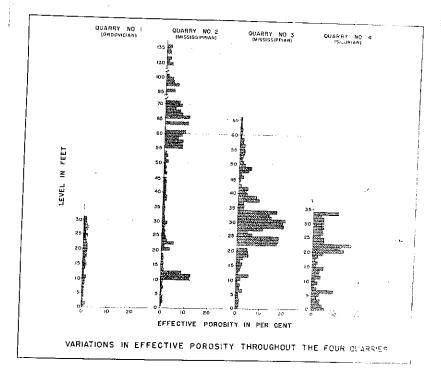
and an attempt was made to classify the individual quarries into zones of sedimentation on the basis of the foregoing tests. The quarries were then chemically analysed zone by zone.

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In Slide No. 6, the variations in amount of insoluble residue on a foot-by-foot basis is indicated for the four quarries tested. Not only does this show that a range in this property occurs in the four quarries, but the quarry averages of Insoluble Residue in percent per unit weight of material could not be correlated directly with the general ratings given the quarry on the basis of pavement performance rating. These averages are: Quarry No. 4, Insoluble Residue 4.166%, Rating - excellent; Quarry No. 2, Insoluble Residue 5.145%, Rating fair; Quarry No. 3, Insoluble Residue 11.900%, Rating - poor.

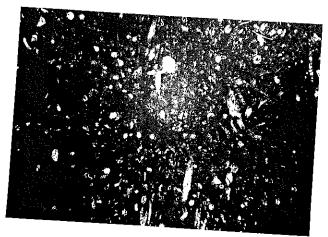




Slide No. 7. Variations in Effective Porosity Throughout the Four Quarries

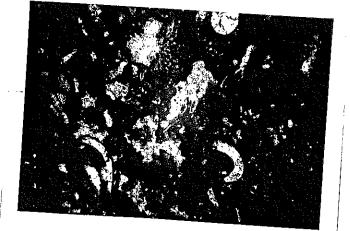
porosity is shown. This again is on a foot-by-foot basis and is intended to show the lack of uniformity within any quarry tested.

Structural variations within a quarry are often extreme. In the following series of slides, a ten foot vertical distance is covered foot-by-foot. These represent thin-sections made from material in Quarry No. 2. In every case the section was made in a vertical plane. The variations within a quarry as shown here is typical for all of the properties thus far investigated.

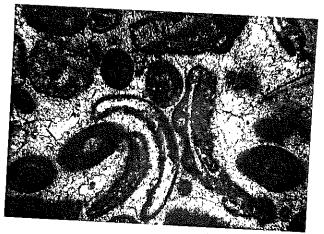


Slide No. 8 61 ft. Level, Quarry No. 2 62 ft. Level, Quarry No. 2





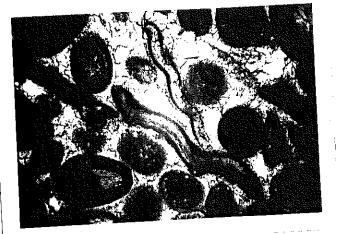
Slide No. 10



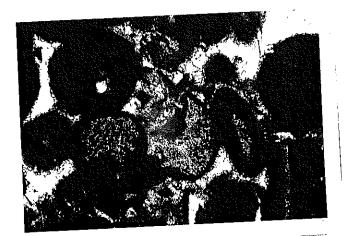
63 ft. Level, Quarry No. 2 64 ft. Level, Quarry No. 2



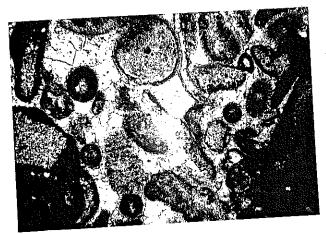
Slide No. 12 65 ft. Level, Quarry No. 2



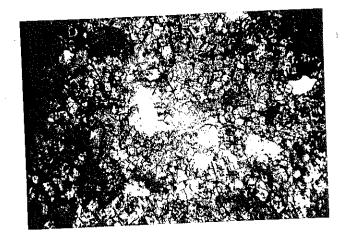
Slide No. 13 66 ft. Level, Quarry No. 2



Slide No. 14 67 ft. Level, Quarry No. 2



Slide No. 15 68 ft. Level, Quarry No. 2



Slide No. 16 70 ft. Level, Quarry No. 2

After the first tests were completed, it became obvious that no two feet would possess exactly the same set of properties. The division of a quarry into zones or ledges was not specific enough. It was decided to sample again for more extensive tests and to locate these specifically as to the exact foot level within a quarry.

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Four exact locations within each quarry were selected. These were chosen for two purposes. First, they were assigned to assure a wide spread in physical and chemical properties. Second, variations within specific geologic formations were considered. Quarries No. 2 and No. 3 include identical geologic formations which can be confirmed, and yet are not at all alike physically and chemically.

In addition to the sixteen levels chosen for test, another location was assigned as a companion to a level from Quarry No. 4. In this location two quarries approximately 1/2 mile apart expose identical formations. The attempt was made to match an exact location in Quarry No. 4 with a sample from the quarry referred to in this project as (JQ).

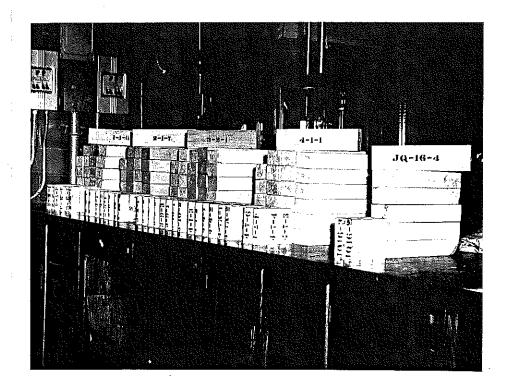
After locations for test purposes were assigned, a careful sampling operation was conducted. The levels designated for test were located and a large block of material removed. The importance of staying within the exact level was stressed.

From these test specimens, beams of limestone were sawed and cores were drilled.

Half of each set were broken, the beams by flexural (third point) break and the cores by compressive break. The remainder were saturated by vacuum and entered in freeze and thaw. Concrete beams were made of the same material using the

fully saturated limestone as both coarse and fine aggregate. Control beams of concrete showed the strength to exceed 700 pounds in every case.

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Slide No. 17. Beams and Cores of Limestone Concrete and limestone beams were measured every seven cycles for length and sonic modulus of elasticity. At the present, durability tests are still in progress so that this phase cannot be reported.

#### PHYSICAL AND CHEMICAL TESTS

The physical and chemical tests which were used to define this limestone are Insoluble Residue, Permeability, Total Porosity, Effective Porosity, Bulk Specific Gravity, Absolute Specific Gravity, Thin Sections, Clay Mineral Content, Los Angeles Abrasion, Toughness, Compressive Strength and Flexural Strength.

Tests under way or planned include Hardness, Size, Shape and Frequency of Pores, Rate of Thermal Expansion and Sodium

and Magnesium Sulfate Soundness. Indications from these tests compared to durability ratings will no doubt indicate further lines of research.

In the early stages of this study, the differences in amount of insoluble residue aroused considerable interest. It was decided to further investigate this fraction.

First, the residues were examined under the microscope. It is necessary for brevity to describe these in generalities. (Slide No. 6)

Quarry No. 1 was highly organic in nature as revealed by insoluble residues. This could be expected from the richly fossiliferous character of the rock. Silica in the form of chert was the most predominate element, often replacing entire fossils. Crystalline Quartz, Galena, Pyrite and Mica formed minor elements in the residues. Clay was prominent in every fraction.

Quarry No. 2 was quite pure, averaging better than 95% carbonates. Silica in the form of sand grains and crystalline quartz was predominant. The minor mineral suites of Pyrite, Mica and Gypsum were present, but in correspondingly smaller amounts. Occassionally small bits of coal could be found. Clay was again present, but not prominent.

Quarry No. 3 was the most impure in composition, with only about 85% carbonates. A peculiar aspect of this quarry was the abundance of large rounded and frosted sand grains. Bearing in mind that Quarries No. 2 and No. 3 are identical in age it is apparent that an abrupt change has occurred in the type of limestone formed. The average amount of residue increased approximately 5 times. In contrast, the minor mineral suites

almost disappeared, with only the occasional appearance of mica to show correlation. Organic material and clay were quite prominent.

Quarry No. 4 was dolomitic limestone with better than 94% carbonates, of which about 18% was MgCO3. The minor mineral suites were dominated by Pyrite, with Quartz and Clay about equally predominant.

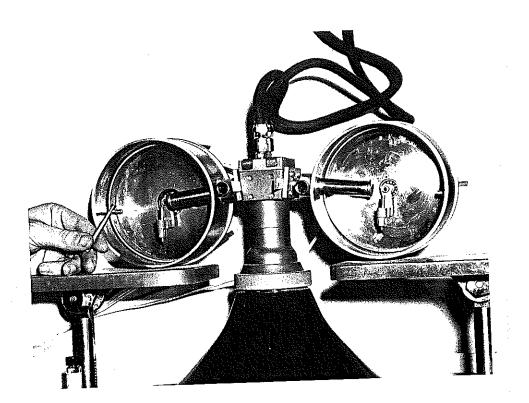
The clay present was analyzed next by quarry zones and next by individual level for the 17 individual levels. X-ray diffraction was employed using the powder method, an adaptation of the Bragg method of X-ray analysis. Clay samples were obtained from a modified insoluble residue process. The colloidal fraction from this process was separated, dried and ground to pass a No. 325 sieve. It was then mounted in a wedge shaped sample holder (shown being held in Slide No. 18), the specimen and holder were then mounted in the Cylindrical Powder Camera. Film was clamped around the inner periphery, the camera sealed, and the specimen was then bombarded with X-rays. The minerals diffracted the X-rays characteristically, and a pattern was obtained on film (Slide No. 19) by which the specimen was identified mineralogically.

In <u>Quarry No. 1</u>, the clay was composed principally of Illite, with one occurrence of pure Kaolinite.

In <u>Quarry No. 2</u>, the clay was composed principally of Illite. However in two instances, there were contrasts. One was a pure Montmorillonite and the other an Illite, Montmorillonite combination.

In <u>Quarry No. 3</u>, the clay was exclusively Illite throughout. In <u>Quarry No. 4</u>, the clay was dominently Illite and Kaolinite.

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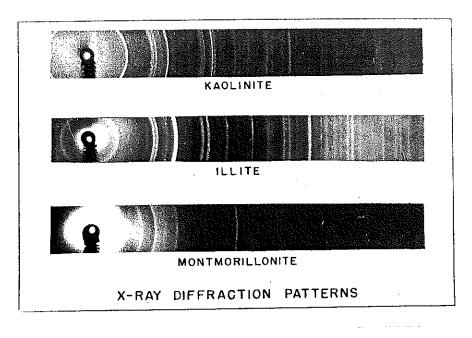
Slide No. 18. X-ray Powder Cameras There were two occurrences of pure Illite.

Future work will be directed towards determinations of the amount of clay mineral present in the residue, and also towards their secondary characteristics such as the cation present.

As yet, no definite correlation between the physical properties have been established by tests on the aggregates themselves. For example: there is no correlation between total void space and thoughness by impact. Also there is no property which may be definitely correlated with general quarry ratings. For example: little difference may be noted between the so-called good quarry and the so-called bad quarry with regard to total void space.

The significance of any of the properties cannot be established until performance tests are completed in the

laboratory. Performance will be determined for several media such as concrete, bituminous mixes and water bound base construction.



Slide No. 19. X-ray Diffraction Patterns