DAH-YINN LEE
FEBRUARY 1970

Progress Report 1
ISU-ERI-AMES-67300

ABSORPTION OF ASPHALT BY AGGREGATE

PROJECT HR-142
IOWA HIGHWAY RESEARCH BOARD
IOWA STATE HIGHWAY COMMISSION
AMES, IOWA 50010

ERI Project 780-S

ENGINEERING RESEARCH INSTITUTE
IOWA STATE UNIVERSITY
AMES, IOWA 50010 USA

ENGINEERING
RESEARCH

ENGINEERING
RESEARCH

ENGINEERING
RESEARCH

ENGINEERING
RESEARCH

ENGINEERING
RESEARCH

PROGRESS REPORT 1

ABSORPTION OF ASPHALT BY AGGREGATE

Dr. Dah-Yinn Lee
Principal Investigator
1 January 1969 to 31 December 1969

Submitted to:
Iowa Highway Research Board
Iowa State Highway Commission

ISU–ERI–AMES–67300
ERI Project 780-S

ENGINEERING RESEARCH INSTITUTE
IOWA STATE UNIVERSITY AMES

1. Report No.
ISU-ERI-Ames-67300

2. Government Accession No.

3. Recipient's Catalog No.

4. Title and Subtitle

ABSORPTION OF ASPHALT BY AGGREGATE

5. Report Date
February 1970

6. Performing Organization Code

7. Author(s)
Dr. Dah-yinn Lee

8. Performing Organization Report No.
ISU-Ames-ERI-67300

9. Performing Organization Name and Address

Engineering Research Institute
Iowa State University
Ames, Iowa 50010

10. Work Unit No.

11. Contract or Grant No.
HR-142

12. Sponsoring Agency Name and Address

Iowa Highway Research Board
Iowa State Highway Commission
Ames, Iowa 50010

13. Type of Report and Period Covered
Progress Report
Jan. 1 – Dec. 31, 1969

14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

This report summarizes work accomplished under Project HR-142 during the period from January 1, 1969 to December 31, 1969.

Pore characteristics of six limestones were studied in relation to their absorption of two asphalt cements. Porosity and pore size distribution were determined by a mercury penetration porosimeter and asphalt absorption was determined by immersion and bulk-impregnated specific gravity methods. A special study was conducted to develop new, simple and more reproducible methods for bulk specific gravity determination. As a result, a new chemical indicator method was developed to determine the saturated surface-dry condition, and a glass mercury pycnometer was designed to determine coarse and fine aggregates. Asphalt absorption history of one asphalt mixture on campus has been studied since September 1968. Chemical and/or radiation treatments of absorptive aggregates were conducted on four chemicals and two aggregates.

17. Key Words
Absorption, asphalt, porosity, pore-size distribution, limestone, aggregate, asphalt paving mixtures, gamma radiation, polymerization.

18. Distribution Statement
Unlimited

19. Security Classif. (of this report)
Unclassified

20. Security Classif. (of this page)
Unclassified

21. No. of Pages
27

22. Price
$3.00

INTRODUCTION

A long-range comprehensive research project entitled "Study of Absorptive Aggregates in Asphalt Paving Mixtures" was initiated in 1967 on evaluation and use of absorptive aggregates for asphalt paving mixtures.

The overall objectives of the study are:

1. To evaluate existing methods and develop new methods for determining both qualitatively and quantitatively, the absorption of asphalt by road aggregates.
2. To correlate basic chemical and physical properties with the absorption characteristics of aggregates with respect to asphalt, and to identify parameters that are indicative of the absorptive characteristics of aggregates.
3. To evaluate the effects of asphalt absorption by aggregate on asphalt, aggregate, and the asphalt mixtures.
4. To establish criteria and tests for identifying, classifying, and specifying aggregates used in asphalt mixtures with respect to asphalt absorption.
5. To develop methods and remedies for utilizing absorptive aggregates in asphalt paving mixtures economically without sacrificing durability and stability of the mixture.

Project HR-127 (1967-1968) was designed to study the first two phases of the overall objectives and, to a limited extent, the third objective. Project HR-142 (1968-1970) was initiated to study the third, fourth, and fifth objectives. This report summarizes the work accomplished during the period from January 1, 1969 to December 31, 1969.

MATERIALS

Six limestone aggregates were studied from quarries selected by Iowa State Highway Commission engineers. The sources and designations of the aggregates are given in Table 1. The locations of the quarries are shown in Fig. 1.

Table 1. Limestone aggregates studied.

No.	County	Quarry	Size (in.)	Beds or Ledges	Geological formation
1-S 1-L	Adair	Menlo	3/8	3-6-Argentine	Missourian series Pennsylvania system
2	Blackhawk	Pints	3/4	Rapid	Cedar Valley formation, Devonian system
3-S 3-L	Hardin	Alden	3/8	-	Gilmore Ciry formation, Mississippian age
4	Scott	Linwood	1/2	Davenport	Devonian system
5-S 5-L	Story	Cook	3/8	-	St. Louis formation, Mississippian series
6	Washington	Keota	1	Beds 14 – 22	Osage series, Mississippian age

Four of the aggregates (Menlo quarry, Adair Co.; Linwood quarry, Scott Co.; Cook quarry, Story Co.; and Keota quarry, Washington Co.) were also studied in HR-127. Limestones from Alden quarry, Hardin County, and Pints quarry, Black Hawk County, are included because of their known service performance records as concrete aggregates. In addition to crushed aggregates from 3/8 to 1 in. size, two "block stones" were obtained from all six quarries for preparation of geometrically symmetrical rock cylinders.

The crushed aggregates were separated by 3/8 in., No. 4, and No. 100

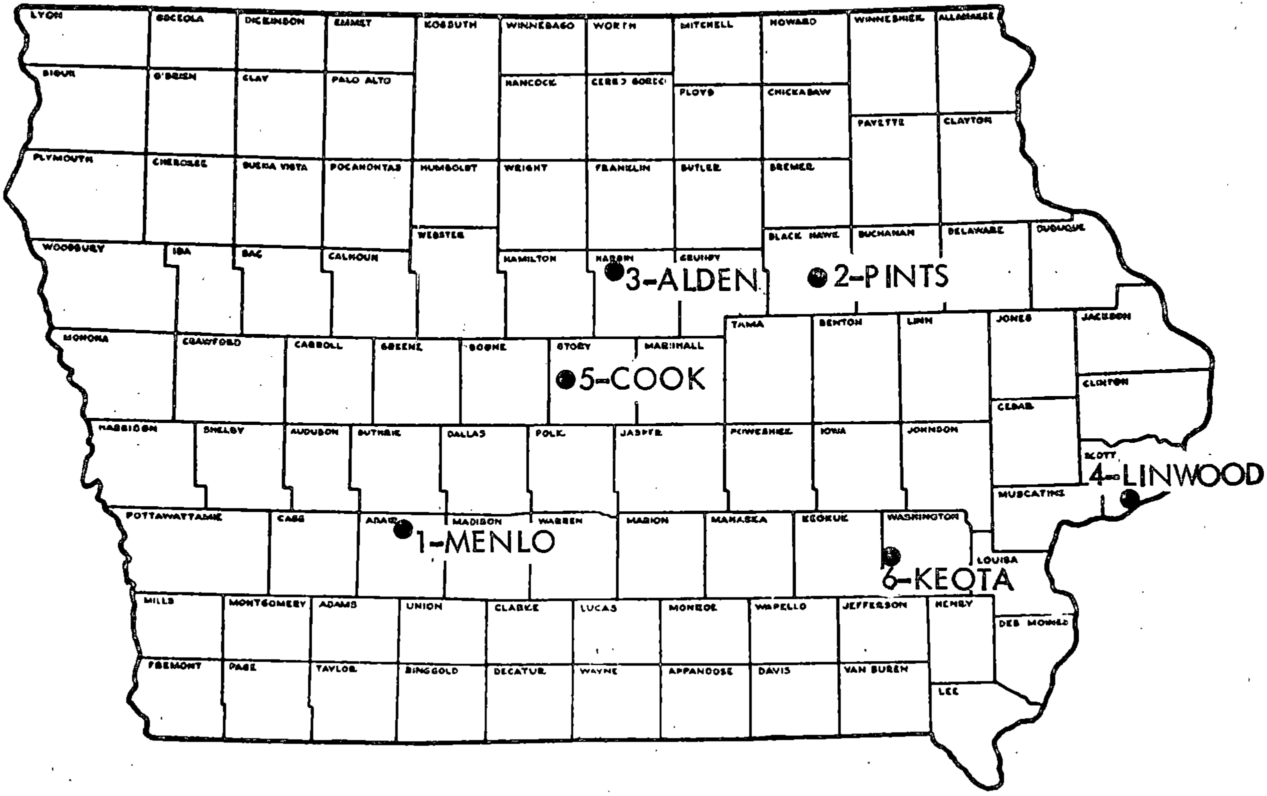


Fig. 1. Project HR-142 aggregate quarry locations.

sieves, washed and oven dried at $110^{\circ}C$ for 24 hours before any tests were conducted.

Various fractions were designated as follows:

A — Retained on 3/8 in. seive

B — Passing 3/8 in. and retained on No. 4 seive

C.— Passing No. 4 seive and retained on No. 100 seive

D — Passing No. 100 seive

D1 — Passing No. 100 seive and retained on No. 200 seive

Two penetration-grade asphalt cements were included in the study. The properties of the two asphalts are shown in Table 2.

Table 2. Properties of asphalt studied.

No.	Property	Asphalt A (85-100 pen)	Asphalt B(120-150 pen)
1	Penetration 77/100/5	92	127
2	Sp. Gr. 77/77	1.008	1.024
3	Flash point, ^{o}F	600	605
4	Fire point, ^{o}F	670	665
5	Softening point, R&B, ^{o}F	120	118
6	Viscosity at 140^{o}F, poises	1144	727
7	Viscosity at 77^{o}F, poises	3.96×10^6	1.58×10^6
8	Thin film oven test		
	% loss	0.10	0.08
	Penetration of residue	54	72
	% retained penetration	57	53
9	Soluble in C Cl4	99.84%	99.83%
10	Ductility at 77^{o}F, cm	130+	130+
11	Spot test	Negative	Negative
12	Percent asphaltenes	17.94	16.93

PORE CHARACTERISTICS AND ABSORPTION

Pore characteristics of both crushed aggregate and rock cores drilled from stone blocks from six quarries were determined to correlate or characterize asphalt absorption with porosity, pore size, and size distribution. Pore properties were determined by a mercury porosimeter.

Pore-size distribution in terms of non-normalized frequency curves of the six rocks are shown in Figs. 2 — 7. Pore-size distribution in terms of relations between cumulative porosity (%) and pore size are shown in Figs. 8 — 13. The phenomenon of hysteresis have been observed in all aggregates, which indicates the existence of ink-bottle pores of varying extent and throat sizes in all rocks. Typical hysteresis curves are shown in Figs. 14 and 15.

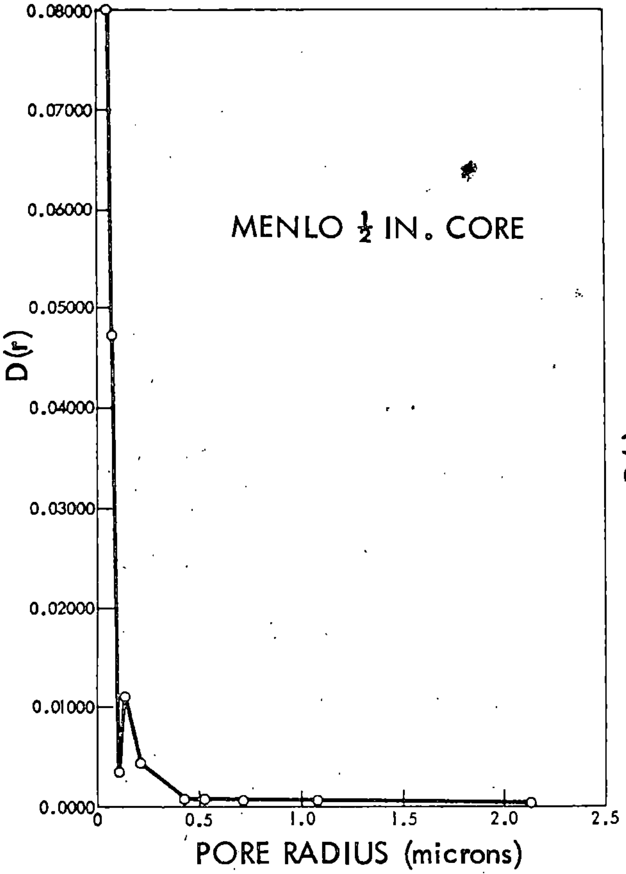


Fig. 2. Non-normalized pore-size distribution (Menlo core).

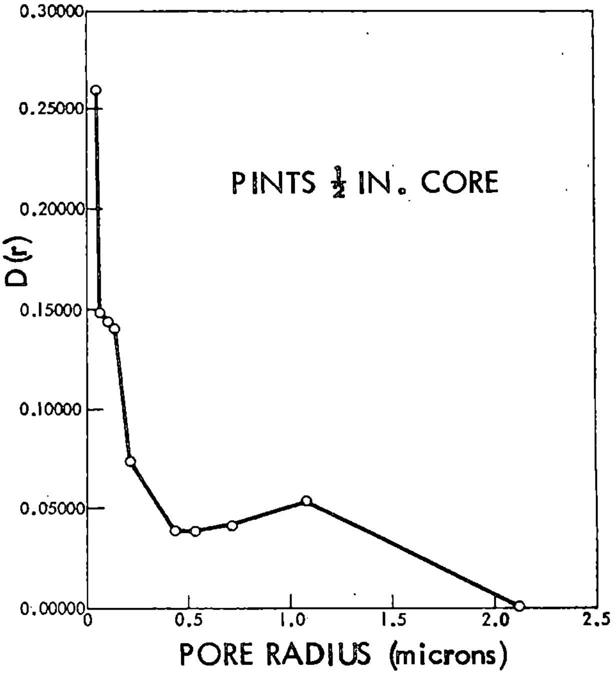


Fig. 3. Non-normalized pore-size distribution (Pints core).

The porosity of the aggregates were determined by the ASTM water absorption method, vacuum saturation method, and mercury penetration method. The results are given in Table 3. Correlation between water and mercury penetration porosities were excellent except for core samples. Variations can be attributed to differences in pore size ranges that were defined by each method, the differences in pore size distributions among aggregates, and poor repeatability of standard bulk specific gravity determinations. However, vacuum-saturated porosity generally gives the highest and mercury penetration gives the lowest porosity results. The correlation coefficient between 24-hours soaked porosity and mercury penetration porosity is 0.7635 (0.9286 without cores); the correlation coefficient between 24-hours and vacuum saturated and mercury penetration porosity is 0.9519, all significant at the 1% level.

Table 3. Effective porosity of aggregates studied.

Quarry	Aggregate No.	Bulk Sp. gr. (ATTM)	Effective porosity, % 24 hour soaked	Vaccuum saturation	Mercury penetration
Menlo	1-S-B	2.567	4.95	4.90	1.94
	1-L-A	2.613	3.08	3.84	1.95
	1-L-B	2.603	3.64	4.22	1.02
	Core	2.637	2.37	–	0.98
Pints	2-A	2.348	15.40	16.91	11.84
	2-B	2.333	17.06	17.80	11.81
	Core	2.271	16.40	–	16.88
Alden	3-S-B	2.475	7.35	8.44	8.24
	3-L-A	2.517	4.88	7.10	6.46
	3-L-B	2.508	5.80	7.55	6.33
	Core	2.510	4.32	–	9.60
Linwood	4-A	2.613	3.72	4.26	2.78
	4-B	2.582	4.29	4.47	2.30
	Core	2.636	2.37	–	0.90
Cook	5-S-B	2.397	14.33	17.74	10.66
	5-L-A	2.426	10.33	13.73	11.54
	5-L-B	2.408	13.00	14.04	11.42
	Core	2.565	5.54	–	17.44
Keota	6-A	2.326	12.98	16.47	10.50
	6-B	2.263	17.00	19.19	14.77
	Core	2.489	7.77	–	4.66

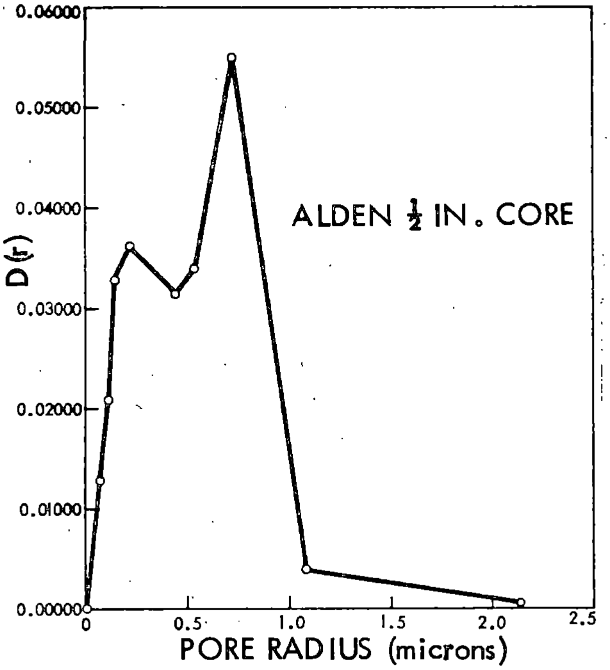


Fig. 4. Non-normalized pore-size distribution (Alden core).

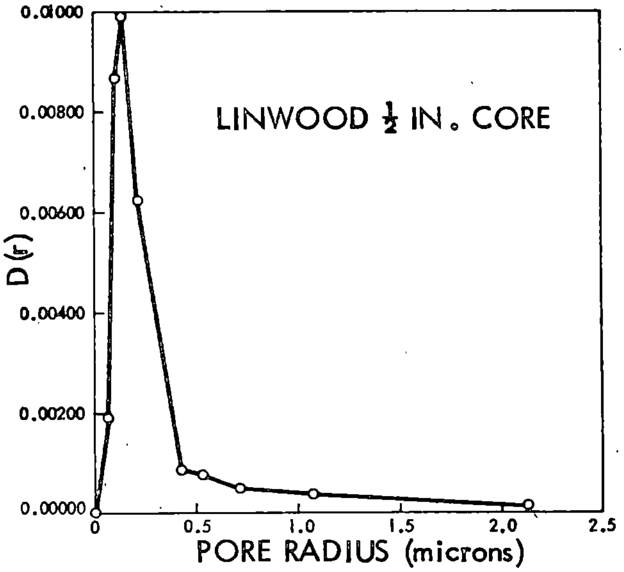


Fig. 5. Non-normalized pore-size distribution (Linwood core).

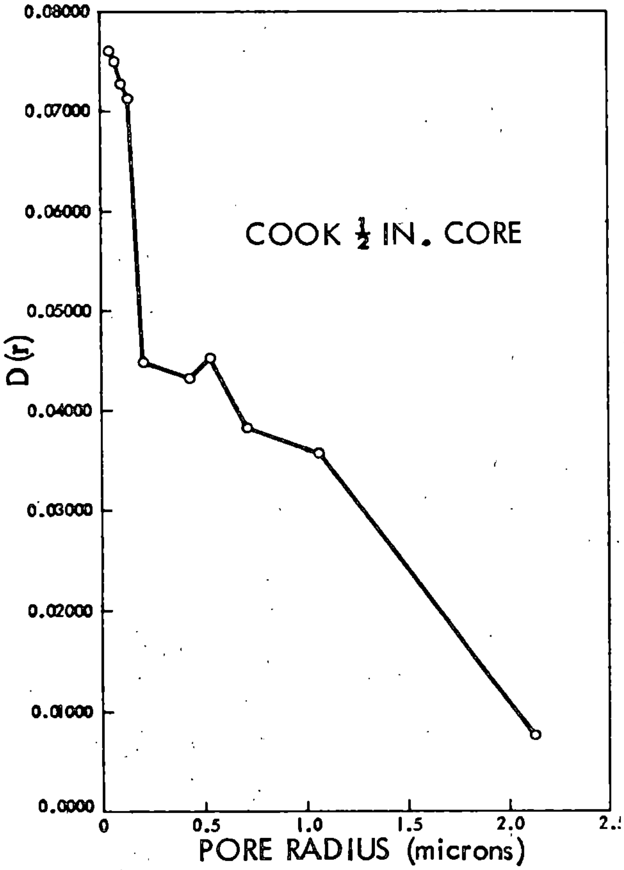


Fig. 6. Non-normalized pore-size distribution (Cook core).

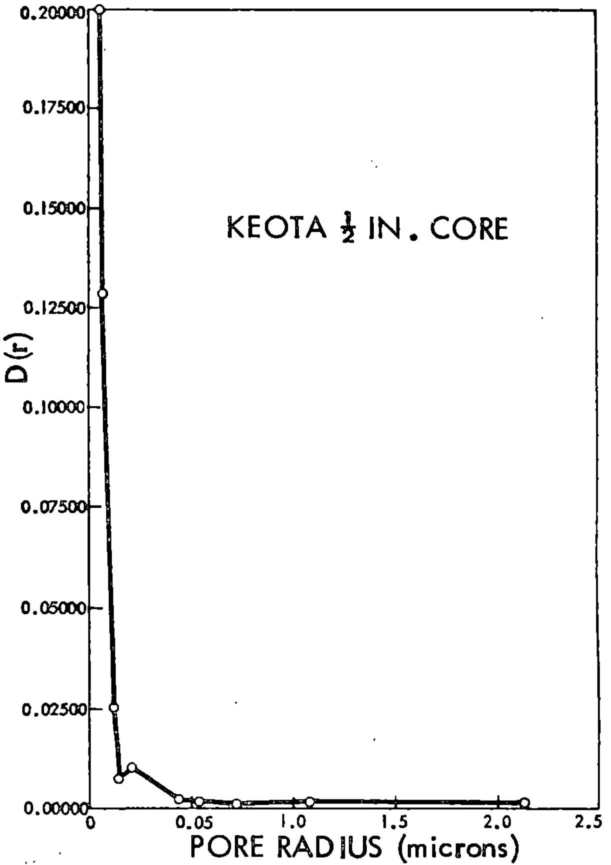


Fig. 7. Non-normalized pore-size distribution (Keota core).

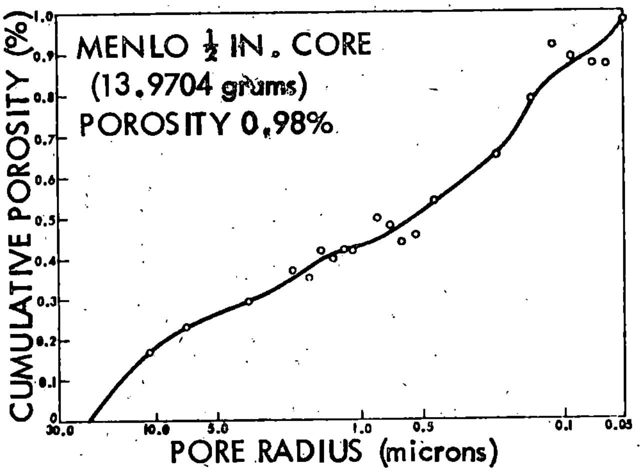


Fig. 8. Cumulative porosity distribution (Menlo core).

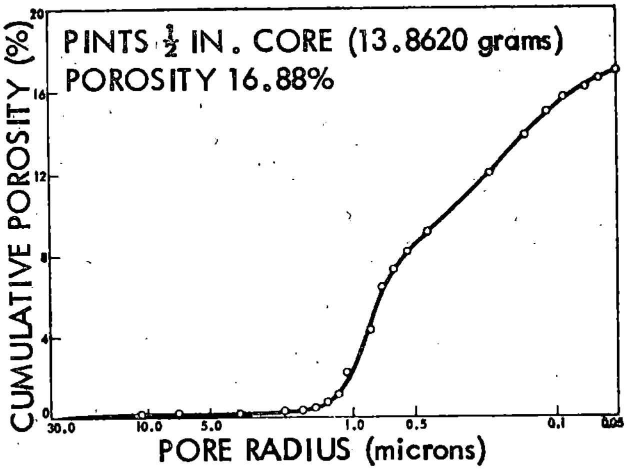


Fig. 9. Cumulative porosity distribution (Pints core).

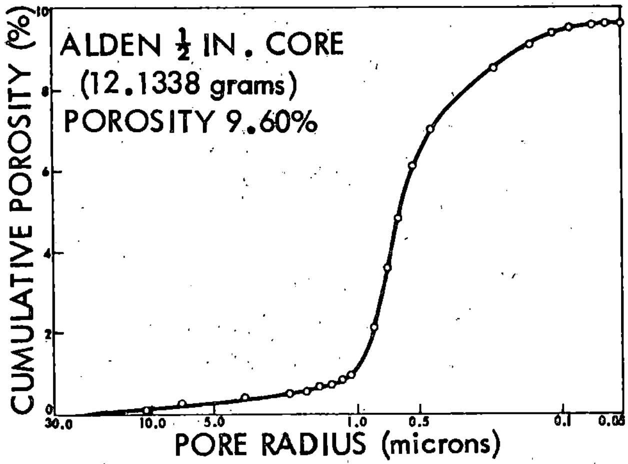


Fig. 10. Cumulative porosity distribution (Alden core).

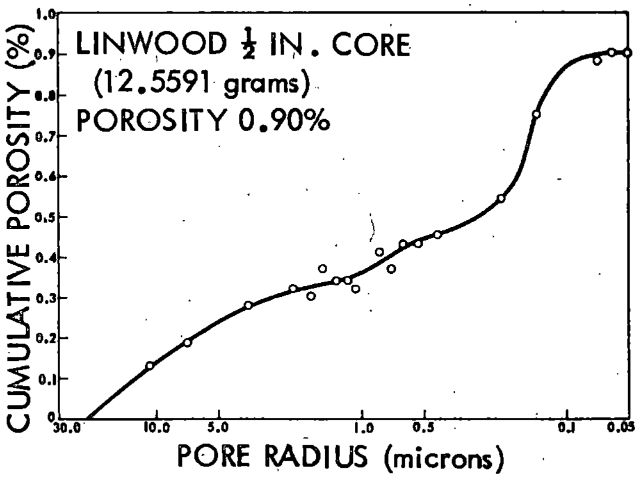


Fig. 11. Cumulative porosity distribution (Linwood core).

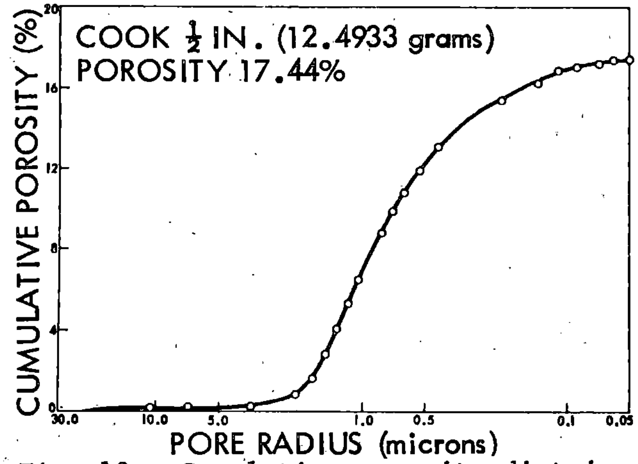


Fig. 12. Cumulative porosity distribution (Cook core).

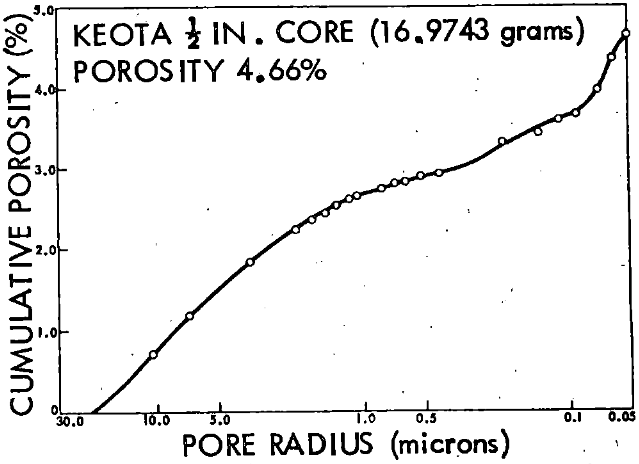


Fig. 13. Cumulative porosity distribution (Keota core).

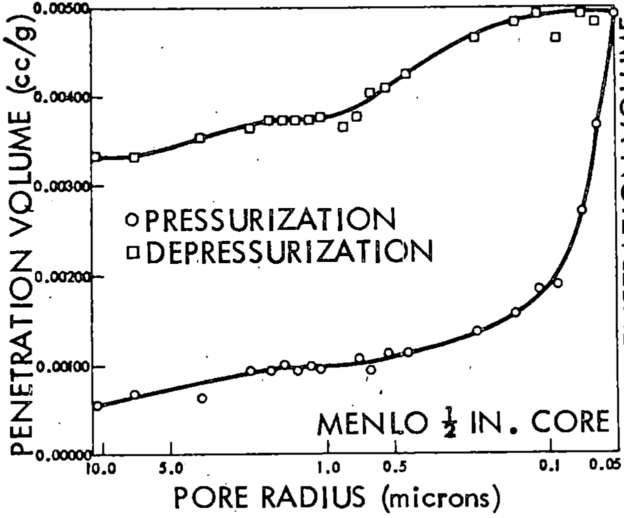


Fig. 14. Hysteresis in mercury porosimetry (Menlo core).

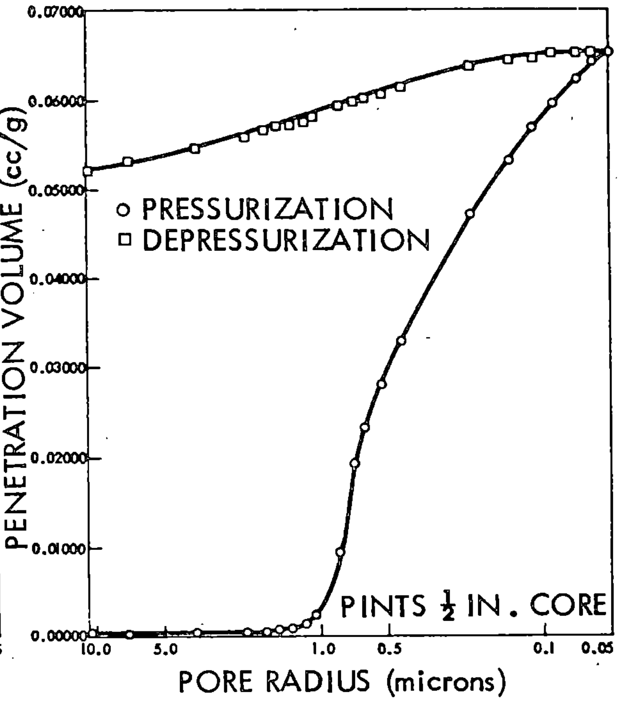


Fig. 15. Hysteresis in mercury porosimetry (Pints core).

Asphalt absorptions were determined by the immersion method and the bulk-impregnated specific gravity method. The results of asphalt absorption by rock core samples are shown in Table 4. Compared with water absorption of the rock cores, the following preliminary conclusions can be stated:

(a) Asphalt absorption by rock cores showed lower values than for crushed aggregates from the same quarries.

(b) The amount and nature of asphalt absorption varies with the method used for absorption determination and the type of asphalt, and

(c) The ratio between asphalt absorption and water absorption varied from about one third to about two thirds. These ratios are controlled by the pore-size distribution of the rocks. The effect of pore-size distribution on asphalt aborption is demonstrated in Fig. 16 in which the cumulative pore-size distribution curves of Menlo and Pints cores are

Table 4. Asphalt Absorption by Rock Cores.

	Absorption, % by core wt.			
	BISG method		Immersion method	
Rock Core	Asphalt A	Asphalt B	Asphalt A	Asphalt B
Menlo	0.30	0.30	0.59	0.59
Pints	2.01	2.27	2.11	2.47
Alden	0.92	2.08	1.20	1.31
Linwood	0.30	0.33	0.44	0.38
Cook	0.84	1.07	1.45	1.83
Keota	0.84	0.96	1.52	1.98

plotted on the same scale. Considerable difference in asphalt absorption between these two types of rocks is reflected by the significant variation between the two curves.

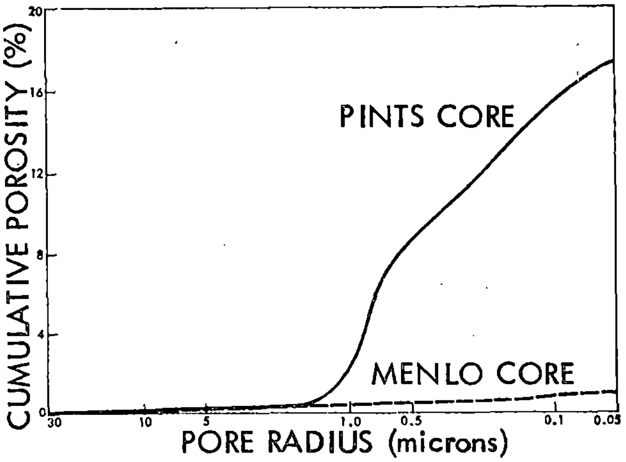


Fig. 16. Cumulative porosity distribution (Menlo and Pints cores).

EVALUATION OF BULK SPECIFIC GRAVITY

Accurate determination of bulk specific gravity of aggregate is of paramount importance in a number of engineering design applications.

Percent voids is generally used as one of the criteria for the design of bituminous paving mixtures. The exact determination of bulk specific gravity of the various constituents of the paving mixture is a necessary part of the design procedure for determination of void properties of bituminous mixtures. It is also used to estimate percent of asphalt absorption by aggregate, which is the major consideration of Projects MR-127 and HR-142. In the design and control of portland cement concrete, bulk specific gravity is used in design calculation of concrete mixtures by the absolute volume concept.

Bulk specific gravity can be defined as the ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. The bulk specific gravity of an aggregate as defined by the ASTM equals the oven-dry weight of the aggregate (A) divided by the sum of the aggregate volume (Vs), the volume of the permeable voids (Vp) and the impermeable voids (Vi), and the unit weight of water (γw):

$$\text{bulk specific gravity} = \frac{A}{(Vs + Vp + Vi)\gamma w}$$

$$\text{or} \qquad = \frac{A}{B - C} ,$$

where B is the saturated surface-dry weight of the material in air, and C is the weight of saturated material in water. Those voids that cannot be filled with water after a 24-hr soaking are referred to as inpermeable voids. Voids that can be filled with water after a 24-hr soaking are referred to as permeable.

ASTM Standards C-127 and C-128 outline methods for determining absorption and bulk specific gravity of aggregates. These standards call for immersion of material in water for 24 hr, followed by drying until the surface-dry state is attained. Coarse aggregates are rolled in an absorbent cloth until all visible water films are gone. Some operators judge this condition by observing the shine contributed by water film while others judge by observing a slight color change in the aggregate.

Fine aggregates are spread on a pan and exposed to a gentle current of warm air until a free flowing condition is reached. The aggregate is then lightly tamped into a conical mold. If the cone stands when the mold is removed, the fine aggregate is assumed to carry moisture on its surface and it is dried further. When the cone just begins to slump upon removal of the cone, it is assumed to be in a saturated surface-dry state.

For natural, well graded fine aggregates, the saturated surface-dry condition is usually reproducible. However, the end point is more erratic for crushed fine aggregates because the angularity of the particles does not permit a definite slump condition as do the rounded surfaces of natural sands. Besides this, the higher percentage of material passing the No. 100 sieve also poses a problem in achieving slump condition.

Various attempts have been made in the past to pinpoint the saturated surface-dry condition of the aggregates to improve the reproducibility of the bulk specific gravity test results. These include Howard's glass jar method[1,2], Martin's wet and dry bulb temperature method[3], Saxer's absorption time curve procedure[4], and Hughes' and Bahramian's saturated air drying method[5]. However, the various modifications either offer little improvement or are too elaborate to be practical in the field or average laboratory.

During the first year of Project HR-142, a special study was conducted to develop new, simple, and more reproducible methods to determine the bulk specific gravity or the saturated surface-dry condition for granular materials. As a result, a new chemical indicator method was developed to determine the saturated surface-dry condition, and a glass mercury pycnometer was designed to determine bulk specific gravity of aggregates larger than the No. 100 seive size. The work on a chemical indicator and evaluation of the method relative to the standard ASTM method was presented at the 1970 annual meeting of the Highway Research Board[6]. Preliminary work on the mercury pycrometer is described in Appendix A.

In the chemical indicator study, the bulk specific gravity of crushed aggregates and cylindrical rock cores from the six HR-142 limestone quarries — one crushed trap rock and one synthetic aggregate (Synopal) from Denmark — was determined by five methods. The methods are: standard ASTM, geometrical measurement, mercury displacement, and two chemical indicator methods (cobalt chloride and fluorescein di-sodium salt) developed at the ISU Bituminous Research Laboratory. The chemical methods are described in Appendix B. The general conclusions of the study were:

1. The ASTM standard tests underestimate the bulk specific gravity of aggregates as these tests measure adsorption as well as absorption of particles. Results obtained in determining bulk specific gravity by other methods such as the mercury displacement method, the geometrical mensuration method, and the chemical methods appear to confirm this statement.

2. Experiments on rock cores indicate that the mercury displacement method gives realistic values between those obtained from the ASTM standard method and the geometrical mensuration method. Further investigations are needed in order to have a properly specified pressure at which measurements of volume displaced by mercury should be taken for consistent results with all aggregates.

3. Chemical methods eliminate the human element to a great extent in observing the saturated surface-dry condition of the aggregates since color change is quite apparent.

4. Attainment of saturated surface-dry condition by the chemical method is not affected by the surface character, particle shape, or gradation of the particles.

5. Results of chemical methods agree well with those obtained from the mercury displacement method.

6. On the basis of the data in this study and the relative cost of each chemical method, the cobalt chloride method appears to be the more preferable of the two chemical methods investigated. However, fluorescein di-sodium salt is better suited when dealing with dark-colored aggregates.

7. Data in this limited study seem to indicate that most duplicate determinations check within 0.01 in the case of the chemical method as compared to 0.02 specified in the ASTM standard test.

8. Additional work is needed in order to establish the reproducibility of the chemical indicator methods.

FIELD STUDY OF AGGREGATE ABSORPTION

As planned in the proposal, field study of absorption effects was to be conducted in the second phase of Project HR-142. It was proposed that four test sections of surface mixtures using absorptive aggregates of varied absorptive capacities (but without compensation of absorbed asphalts) would be laid as parts of regular construction projects during the 1968 construction season and the following parameters would be studied:

1. Nature, occurrence, and frequency of cracks that may be observed at six-month intervals.
2. The absorption history determined by the Rice method from plant to pavement and at six-month intervals.
3. Hardening and chemical changes in the asphalt at six-month intervals.

On August 6, 1968, Mr. Stephen E. Roberts, Research Engineer of the Iowa State Highway Commission, sent letters to nine county engineers (Butler, Decatur, Dubuque, Floyd, Mahaska, Sac, Wapello, Webster and Hardin Counties) asking for cooperation in the program. Subsequently, on September 11, 1968, letters were sent to the same county engineers by the principal investigator of Project HR-142, explaining the detailed field study program proposal. While some of the counties responded, none could participate in the program either because it was too late in the season or because no absorptive aggregates would be used in their paving projects. It is hoped that field study can be commenced during the 1970 construction season on either state or county paving projects.

Nevertheless, absorption history has been studied on one asphalt surface project at Iowa State University laid on September 28, 1968. Aggregates used for the project were blends of 70% Cook's quarry 3/8-in. limestone and

30% concrete sand. Upon extraction, it was found that the asphalt content was 8.5% and the gradation of the aggregate as shown in the following table.

About 20 lb of the mixture was collected at the paver on September 28, 1968. Rice specific gravity of the mixture has beed determined at regular intervals. Calculated asphalt absorption up to 368 days is shown in Fig. 17, which clearly shows the increased asphalt absorption at a decreasing rate up to a year. This observation will be continued.

Sieve	Percent Passing
3/8 in.	99.0
No. 4	77.9
No. 8	61.5
No. 30	29.9
No. 50	13.0
No. 100	6.9
No. 200	6.3
Bulk Specific Gravity	2.374

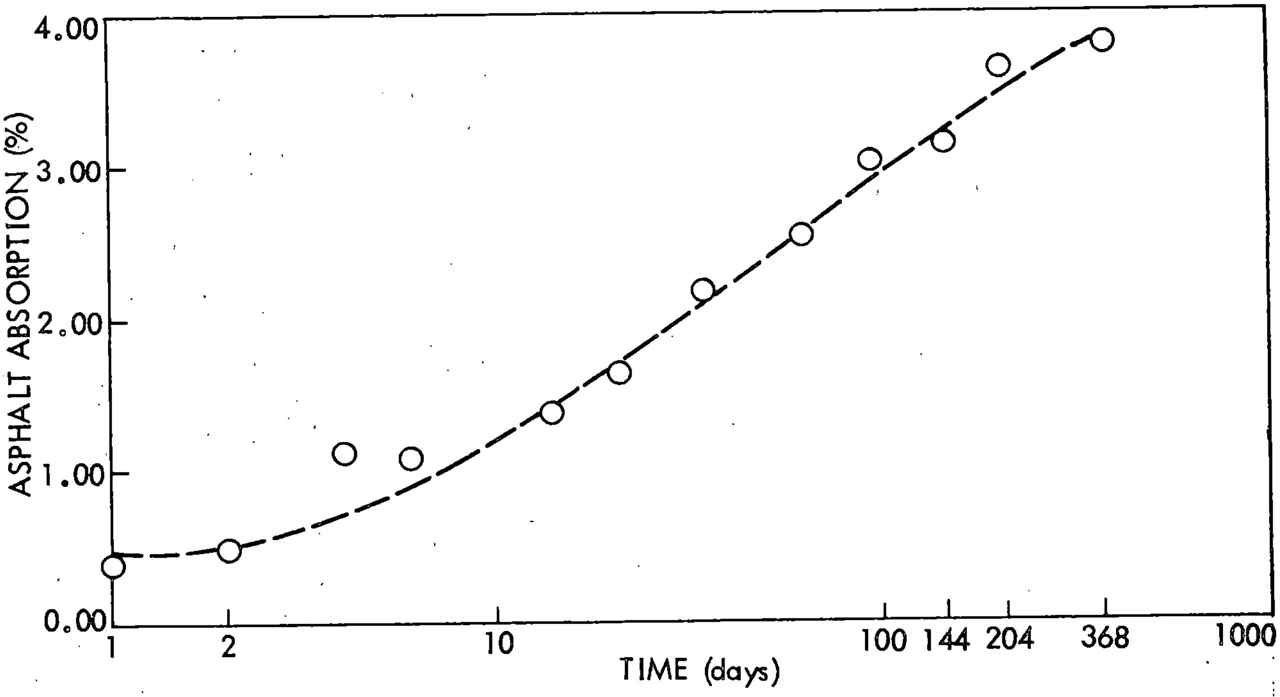


Fig. 17. Asphalt absorption vs. time.

CHEMICAL TREATMENT OF ABSORPTIVE AGGREGATES

Preliminary laboratory investigation on treatment of absorptive aggregates has started during the first year using several chemicals. The purpose of the treatment is to reduce absorption by sealing aggregate pores. Four chemicals were studied, based on previous work on soil stabilization and concrete durability. The chemicals are:

1. 2:1 Aniline/Furfural
2. Armac T (acetic acid salt of a tallow primary amine)
3. Type "N" sodium ailicate
4. Methyl methacrylate

Graded aggregates from Cook and Menlo quarries passing the 3/4-in. sieve and retained on the No. 8 sieve were treated with different percentages of 2:1 aniline/furfural (3 to 9%, Armac T (1 to 6%) and sodium silicate (5 to 10%). Water and asphalt absorptions were then determined on the treated aggregates, using ASTM C-127 and bulk-impregnated specific gravity methods. It was found that, in the case of 2:1 aniline/furfural and Armac T, 6% treatment was necessary for effective reduction of water and asphalt absorption and adequate heat stability (400^{o}F for 4 hours). For sodium silicate treated aggregates, no appreciable reduction in absorption resulted. The mechanisms and modifications of these treatments will be studied further.

One-inch diameter rock cores from Menlo and Cook quarries were used to study methyl methacrylate monomer treatment. Cores were loaded under vacuum with the monomer solution (with 1% benzol peroxide and 10% trimethyl propane trimethacrylate to speed up the polymerization process and to increase the softening point of the polymer) and then polymerized. Polymerization was accomplished by thermal treatment (two hours at 185^{o}F), and gamma irradiation (cobalt 60 radiation at a dose rate of 0.6 megarad per hour to a total dose of 1 megarads). Gamma irradiation was done at the Gamma Irradiation Facility in the Ames Laboratory Research Reactor. As much as 100% reduction of water and asphalt absorption was obtained by both methods.

Treatments of graded aggregates are being evaluated on asphalt mixtures. Aggregates meeting Iowa Type A asphalt concrete gradation were treated with chemicals, and asphalt concrete mixtures were prepared and compacted by the Marshall method. Stability, flow, absorption, and immersion-compression tests are being conducted on treated mixtures. Comparison will be made with mixtures of untreated aggregates to evaluate the effectiveness of these chemicals in reducing water and asphalt absorption.

Based on the results of methl methacrylate treated aggregates, plans are being made to determine the effectiveness of cheaper monomers such as styrene, arcylonitrile, styrene-acrylonitrile, vinyl acetate, ethylene, etc.

FUTURE WORK

The following work will be undertaken during the next 12 months of Project HR-142:

1. Refine and improve existing and developed methods of bulk specific gravity and asphalt absorption tests.

2. Initiate with the Iowa Highway Commission's cooperation the field study phase of the program.

3. Continue study on treatments of absorptive aggregates in the laboratory.

4. Plan and propose a field feasibility study of using absorptive aggregates in asphalt paving mixtures by laboratory developed techniques.

ACKNOWLEDGMENT

This is a progress report of a study under Project 780-S of the Engineering Research Institute, Iowa State University, under the sponsorship of the Iowa Highway Research Board and the Iowa State Highway Commission.

The author wishes to express his thanks to Messrs. Steve Roberts, Bernard Ortgies and James Young, all of the Iowa State Highway Commission, for their cooperation; to Dr. John Lemish, Department of Earth Science, for permission to use the mercury penetration porosimeter; to Dr. Adolf Voigt, Ames Laboratory, for permission to use the Gamma Irradiation Facility; and to Mr. Joseph Crudele, Ames Laboratory, for helping with radiation dosimetry work.

REFERENCES

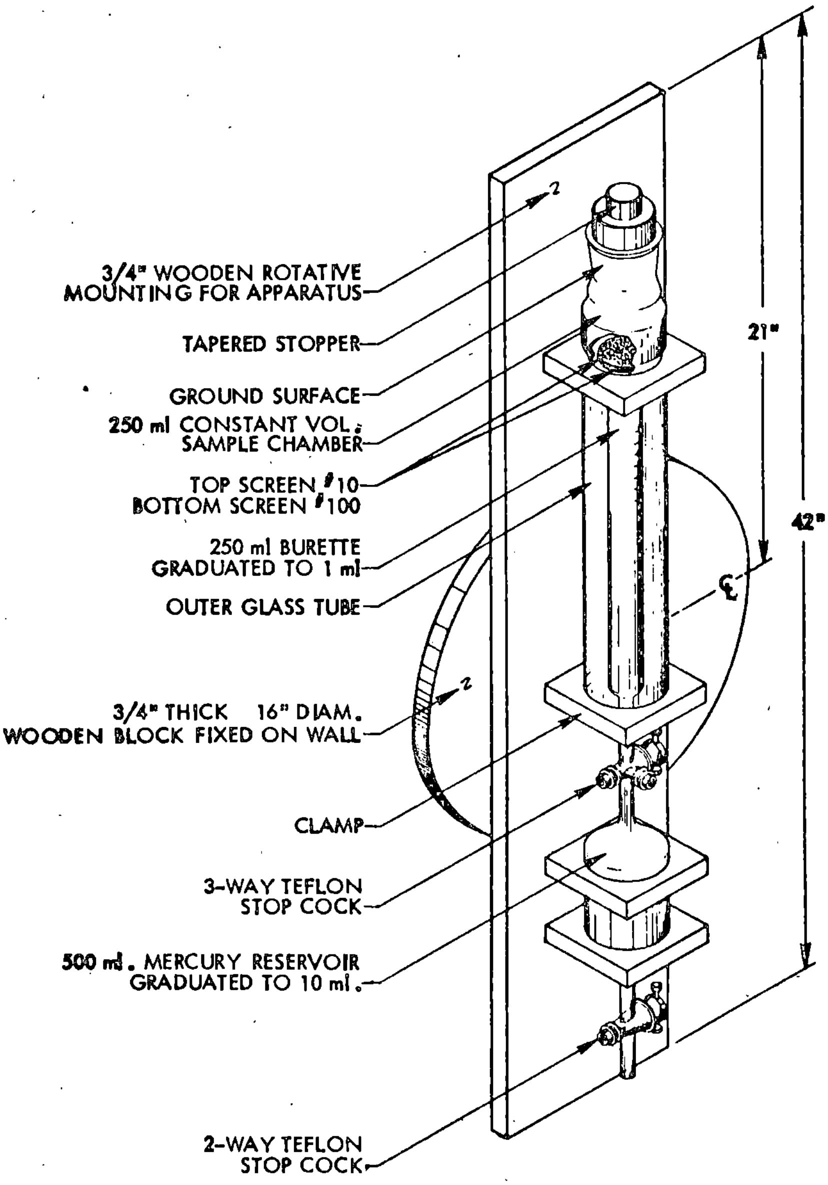
1. Howard, E. L., discussion, Proc. American Concrete Institute, 54, 1215 (1958).
2. Pearson, J. C., "A Simple Titration Method for Determining the Absorption of Fine Aggregate," Rock Products, 32, 64 (1929).
3. Martin, J. Rogers, "Two Years of Highway Research at Oklahoma A&M University," Proc. AAPT. 19, 41 – 54 (1950).
4. Saxer, E. L. "A Direct Method of Determining Absorption and Specific Gravity of Aggregates," Rock Products, 87, 77 – 79 (1956).
5. Hughes, B. P. and B. Bahramian, "An Accurate Laboratory Test for Determining the Absorption of Aggregates," Materials Research and Standards, pp. 18 – 23 (1967).
6. Kandhal, P. S. and D. Y. Lee, "An Evaluation of the Bulk Specific Gravity for Granular Materials," paper presented at annual meeting of the Highway Research Board, Jan. 12 – 16 (1970), Washington, D.C.

APPENDIX A

Mercury Pyconometer for Determination of Bulk Specific Gravity

Description

The sketch of the apparatus dimensioned in detail is shown in the figure below.



MERCURY PYCNOMETER FOR DETERMINATION OF
BULK SPECIFIC GRAVITY OF AGGREGATES

The apparatus consists of: (a) 250 ml constant volume sample chamber
(b) 250 ml burette
(c) 500 ml mercury reservoir.

The apparatus is mounted on a rotative wooden mounting which rotates on a pivot provided in a circular wooden block fixed to the wall.

Procedure

Mercury is filled in the mercury reservoir with the apparatus in an inverted position (with 3-way stop-cock closed). The apparatus is then set in the normal position. The following steps comprise a normal determination:

1. After applying vaccum grease, the stopper is inserted, and secured with clamps (or springs) in place.
2. Apply the 27-in. vacuum and evacute the apparatus for 10 min.
3. Rotate and invert the apparatus. Record Hg level in the burette (A,cc) at 1 atm.
4. After closing the 2-way stop-cock and evacuating the apparatus again, rotate it and bring it to its normal position.
5. Open the 3-way stop-cock to "atm." Open the stopper and put the prebiously weighed (W, gms) aggregate in the sample chamber. Replace the stopper and secure it tightly to the apparatus.
6. Evacuate the apparatus for 10 min.
7. Rotate and invert the apparatus; record the displaced Hg level in the burette (B, cc) at atm pressure.
8. Close the 2-way cock, evacuate the apparatus for 10 min., then bring it to its normal position.

$$\text{Bulk specific gravity} = \frac{W}{B - A}$$

9. The mercury should be cleaned occasionally. This can be done by putting mercury in a container, covering it with a layer of 10% nitric acid, then blowing air through it by applying a vacuum for 3-4 hours, and then washing it with water and drying with blotting paper.

Data

A preliminary test was conducted on 1/2-in. rock cores, and the following results were obtained:

Rock Core	Bulk specific gravity by pyconometer	Bulk specific gravity by ASTM
Menlo	2.710	2.637
Pints	2.312	2.271
Linwood	2.694	2.636
Cook	2.622	2.565
Keota	2.549	2.489

Samples were weighed to 0.0001 gm and volume observed to 1/4 cc.

Improvements

The following improvements are suggested:

1. A more effective clamp is required to ensure no movement of the stopper.
2. To improve the apparatus, the following could be done:
    (a) Have the burette read to the nearest 1/10 ml instead of 1/4 ml as on the present apparatus.
    (b) The orifice at the bottom of the sample chamber (below the screens) could be widened to have a bore equal to that of the burette. This is necessary since the present apparatus has to be rotated back and forth to eliminate residual air bubbles trapped between the orifice and fine screen.

(c) Good stop-cocks to withstand the vacuum; the present ones, though teflon coated, have to be coated with vacuum grease.

3. Contamination of the mercury by grease in the stopper of the sample chamber and stop-cocks should be avoided.

APPENDIX B

Determination of Bulk Specific Gravity of Aggregates by Chemical Indicator Methods

Cobalt Chloride ($COCl_2$) Method

The procedure is the same as for ASTM C-127 and C-128 test except for the following points:

1. The sample is immersed in a 5% solution of cobalt chloride instead of plain water for 24 hr.
2. The sample is removed from the solution and put on a white surface such as a table or enameled tray. Aggregate may not seem colored, but the visible water films on the aggregate will be pinkish in color.
3. The sample is spread to have a layer of individual particles exposed to a gently moving current of warm air. It is stirred frequently to secure uniform drying. Some of the solution sticking to the porcelain will appear to be pink.
4. As drying proceeds, the aggregate attains bluish color which helps in adjusting the warm air. Aggregate should be turned over frequently by gentle hand action. If there is still some moisture left on the surface of the aggregate, the bluish color on the porcelain will change back to pink. Keep turning the sample, while exposing it to warm air, until all the aggregate is bluish in color and the bluish spots on the white porcelain no longer turn pink. This is assumed to be saturated surface-dry condition.

5. The saturated surface-dry coarse aggregate is immediately weighed in air and water as outlined in the ASTM C-127 test, while the fine aggregate is introduced into the Chapman's flask, which has been filled with water to the 200 ml mark.

Fluorescein Di-Sodium Salt (FSS) Method

The procedure is essentially the same as the $CoCl_2$ method. In this case a 0.5% solution of fluorescein di-sodium salt is used.

The sample is dried in the same manner as in the previous test. The sample acquires a yellowish color when taken from the solution after 24 hr immersion. On drying, an orange color appears on the aggregate, the porcelain also changes to a distinct orange. This is assumed to be the saturated surface-dry condition and the sample is further treated as per ASTM C-127 and C-128 tests' procedure.

The $CoCl_2$ method is suitable for whitish to light grey colored aggregate. For dark aggregate, a 10% solution of $CoCl_2$ can be employed or FSS can be used.

Before removing the samples from the solution, the latter should be agitated by turning over the aggregate.

Because a bluish color acquired by the saturated surface-dry aggregate fades with time, indicating possible adsorption of moisture from air or drawing moisture from within through capillaries, the sample should be weighed immediately.

JK/ks