

# HIGHWAY RESEARCH RECORD

**Number 307**

## Synthetic Aggregates and Granular Materials

5 Reports

### Subject Areas

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| 32 | Cement and Concrete            |
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## Foreword

Of all the materials used in the highway system, aggregate by far represents the largest quantity hauled to the job. Hundreds of millions of tons are utilized annually for all types of purposes, including high-quality, skid-resistant aggregates for surfaces, aggregates for portland cement concrete and asphaltic concrete, and aggregates for bases and subbases. These needs place tremendous demands on the nation's supply of this resource. Coupled with this demand is a strong need to better characterize the aggregates so that they may be utilized more effectively.

As a result of the tremendous use of aggregates and the rapid urbanization of our population, natural aggregates are being rapidly depleted in many areas of the country. Thus, a demand is being created for suitable aggregate replacements, and the materials engineer must look to new sources of materials to supply this demand. In this RECORD four reports deal with such aggregate replacements, termed "synthetic" aggregates. Malisch, Day, and Wikson deal with domestic waste glass used as an aggregate in bituminous concrete. Research results indicate that bituminous concrete mixes made entirely with domestic waste glass as the aggregate can be designed to satisfy the Marshall design criteria. Another aggregate replacement described by Hendrickson and Lund is volcanic cinders. The successful utilization of these cinders as a wearing surface for unpaved forest access roads is fully documented. A third synthetic aggregate discussed is sintered fly ash, and Minnick's paper describes the properties and performance of this material when made into a structural lightweight aggregate for portland cement concrete. Information is also presented on typical mix designs for use in structural concrete. A fourth synthetic aggregate, described by Moore, is that produced in a rotary kiln from raw clay. This aggregate is nonbloated and similar in properties to brick particles. Research results, coupled with field evaluation, indicate that this material performs very well as an aggregate in flexible bases.

Kandhal and Lee discuss the improved characterization of aggregate, in particular the bulk specific gravity. The standard ASTM methods for determination of a saturated surface-dry specific gravity for both coarse and fine aggregates are compared with five other methods. Results indicate that the standard ASTM tests underestimate the bulk specific gravity and may not be as reproducible as other methods suggested.

—W. B. Ledbetter



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# Use of Domestic Waste Glass as Aggregate in Bituminous Concrete

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University of Missouri—Rolla

The increasing amounts of waste glass being generated in the United States are necessitating the development of new methods for disposal of this refuse component. The use of waste glass as an aggregate in bituminous concrete was investigated in this study. The objectives were to determine whether a mixture using glass aggregate could be designed that would meet Marshall test design criteria, to investigate the amount of degradation occurring in these mixtures, and to determine the water-resistance of such mixtures.

Waste glass was crushed and screened into several size fractions, which were then combined to give a maximum density gradation. Standard Marshall design methods were used to determine stability, flow, and void parameters at several asphalt contents, and to determine an optimum asphalt content satisfying Marshall design criteria. Extraction tests were conducted on some of the specimens tested, and mechanical analyses of the recovered aggregate were used to assess the amount of degradation of the aggregate. Static stripping tests were conducted on several combinations of glass aggregates and bituminous materials.

Bituminous mixtures satisfying Marshall design criteria can be designed using aggregates composed entirely of crushed glass. Some degradation of the glass aggregate does occur under laboratory mixing, compacting, and testing conditions, with greater degradation appearing to be associated with gradations deviating from those giving maximum density. Severe stripping occurs when the glass-aggregate bituminous concrete with no antistripping agent is exposed to water. Glass aggregates coated with asphalt cements containing antistripping agents or coated with cationic emulsions do not strip in the static stripping test.

•SOLID WASTES generated in the United States each year total 3.5 billion tons according to the U. S. Public Health Service. This waste includes about 30 billion glass bottles and jars, with increasing amounts of this component expected as the use of "one-way" bottles continues to increase at a rapid rate (1). Glass is a particularly troublesome component of waste because it will not burn, rust, or decay. A study of the composition and characteristics of municipal incinerator residues (2) indicates that glass constituted the major fraction of incinerator residues and accounted for nearly half of the residue by weight. Thus, even after incineration, there is still a considerable amount of glass remaining to be disposed of.

Other means of disposal of glass include sanitary landfills and salvage and reclamation operations. In some areas, sanitary landfills are doubtful solutions to the problem because of the lack of available land. As these landfills are moved further and further from the urban centers, transportation costs mount and this method becomes less

appealing. The bulk of glass is also a problem because bottles take up an inordinate amount of space in relation to the glass present, unless the bottles are thoroughly crushed.

An alternate method of disposing of waste glass is to reuse the material, perhaps in a different form. Economic considerations have generally dictated whether or not salvage and reclamation operations are feasible, and, in the past, processing and transportation costs have usually ruled out this possibility. For instance, waste glass could be returned to a glass manufacturer to be melted and reused, but transportation costs and the costs of grading the glass into different categories by color, type, and so forth, are generally prohibitive. Furthermore, particularly in large cities, public works officials are justifiably apprehensive of reliance on a disposal method that depends on fluctuating and sometimes nonexistent markets for salvage by-products. If the markets collapse, the city may find itself with quantities of refuse and no method of disposal except on an emergency landfill distant from the city (3). Thus, in order to maximize potential benefits from salvage and reclamation operations, it is necessary to minimize processing and transportation costs while ensuring a steady market for the salvaged refuse.

By using waste glass as aggregate in bituminous concrete mixtures for maintenance of city streets, transportation costs would be minimized because the glass would be used in the city where it was discarded. Separate collections of glass refuse, although increasing collection costs, would make costly hand-sorting unnecessary, and all processing (cleaning, crushing, and screening) could be mechanized. A steady market would be available because of the continuous street maintenance programs of cities, and the savings from purchasing less aggregate would partially offset the increased costs associated with separate collections. A final benefit from this utilization of glass as an aggregate may materialize later as present sources of conventional aggregates are depleted and new sources are sought.

### OBJECTIVES

This paper describes work done in investigating the use of crushed waste glass as an aggregate in bituminous mixtures. No previous investigations of this nature could be found in the literature, and therefore the following initial objectives were defined:

1. Design a dense-graded bituminous mixture using glass aggregate that would meet Marshall design criteria specified by The Asphalt Institute;
2. Determine the amount of degradation that occurs during laboratory mixing, compaction, and testing of the mixtures; and
3. Determine the water-resistance of the mixtures.

### MATERIALS

All aggregates used in this investigation were obtained by crushing waste glass consisting primarily of one-way beer and soft-drink bottles. The bottles were first washed in hot water to remove labels and any other foreign material. After drying, they were passed through first a jaw crusher and then a roller mill for finer crushing, after which the crushed material was dry-sieved into nine different size fractions ranging from material passing a  $\frac{1}{2}$ -in. sieve and retained on a  $\frac{3}{8}$ -in. sieve to material passing the No. 200 sieve. A washed sieve analysis was performed on samples from each of the size fractions, the results of which were used in combining the fractions to obtain a desired gradation.

The crushed material contained a relatively large amount of flat or elongated particles or both in nearly all fractions. Samples from several of the larger size fractions were tested for percentage of flat and elongated particles using Corps of Engineers Methods CRD-C 119-53 and CRD-C 120-55. Each size range sample was subdivided until approximately 100 to 300 particles were obtained, with the larger sample size being used for smaller particles. The length, width, and thickness of these particles were measured and the particles were classified according to the ratios of length to width and width to thickness. A flat particle had a width-to-thickness ratio of 3 or greater, whereas an elongated particle had a length-to-width ratio of 3 or greater. Results of

TABLE 1  
FLAT AND ELONGATED PARTICLE COUNT

Sieve Size		Particles Counted	Percent in Each Class			
Passing	Retained		Flat <sup>a</sup>	Elongated <sup>b</sup>	Flat and Elongated	Not Flat or Elongated
1/2 in.	3/8 in.	101	93	0	4	3
3/8 in.	No. 4	117	48	3	2	47
No. 4	No. 8	300	9	19	0	72
No. 8	No. 16	306	25	2	0	73
No. 16	No. 30	305	49	3	1	47

<sup>a</sup>Width to thickness ratio greater than 3.0.

<sup>b</sup>Length to width thickness ratio greater than 3.0.

these particle measurements are given in Tables 1 and 2. As was expected, nearly all particles in the 1/2- to 3/8-in. size range were flat, but the percentage of flat and elongated particles decreased as the size of the sieve opening on which the particles were retained approached the wall thickness of the bottles. In the material passing the No. 16 sieve, the percentage of flats began to increase again, and microscopic investigation of the material finer than the No. 30 sieve indicated the presence of a significant number of flat and elongated particles. Photomicrographs of the finer fractions are shown in Figure 1.

Specific gravity and Los Angeles abrasion tests (gradation C) were conducted on the glass, and a hydrometer analysis was conducted on the material passing the No. 200 sieve. The bulk specific gravity of the crushed glass aggregate was 2.50, the absorption was 0.01 percent, and the Los Angeles abrasion loss was 41 percent.

The asphalt used was an 85-100 penetration asphalt cement furnished by the Shell Oil Company and produced from a West Texas crude. Its properties are given in Table 3.

### TEST PROCEDURES

Marshall test procedures, as specified by ASTM Method D 1559, were followed with the following exceptions:

1. Immediately after mixing the specimen in a Hobart Model N-50 mixer for 2 minutes, the bituminous mixture was placed in the compaction mold and then the mold was placed in an oven maintained at 275 F for 30 minutes. It was then removed and spaded and compacted as specified in ASTM Method D 1559.

2. In the first trial mix series, rather than placing the specimens directly in a 140 F water bath for 30 minutes prior to testing, they were first placed in a plastic bag and then in the water bath. This was done to prevent surface stripping, which would alter the results of mechanical analyses of the extracted aggregate. Some leakage occurred in most of the bags, however, and in the second trial mix series all specimens were placed in an oven at 140 F for 2 hours prior to testing.

After the completion of stability testing, bitumen content was determined using a reflux extractor, and a mechanical analysis of the extracted aggregate was performed using AASHTO Method T 30.

TABLE 2  
SIZE DISTRIBUTION OF MINUS-200 MATERIAL

Particle Size (microns)	Percent Finer	Particle Size (microns)	Percent Finer
75	100	20	17
60	82	10	6
40	50	5	2

TABLE 3  
PROPERTIES OF ASPHALT

Property	Value
Specific gravity at 60 F	1.011
Penetration at 77 F	92
Viscosity, Saybolt Furol at 275 F, sec	143.5
Flash, Cleveland open cup, deg F	595
Solubility in CCl <sub>4</sub> , percent	99.9
Ductility at 77 F, cm	150+

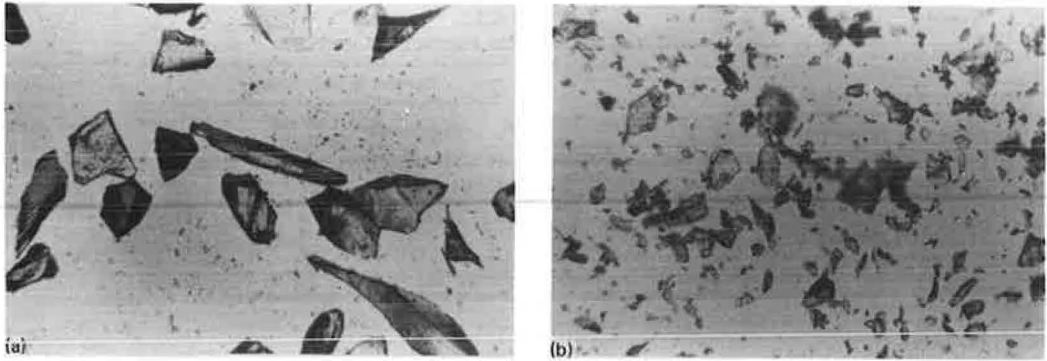


Figure 1. Photomicrographs showing particle shape of fine crushed glass fractions: (a) plus-100 mesh size fraction (X 27), and (b) minus-200 mesh size fraction (X 67).

MIX DESIGN

The first trial mix used aggregate gradation No. 1 as given in Table 4. This gradation is one suggested by Goode and Lufsey (4) for maximum density and is calculated from the relationship

$$P = \left(\frac{d}{D}\right)^{0.45} \times 100$$

where P = percent passing a sieve having an opening of d inches,  
D = maximum size of the aggregate, and  
0.45 = an empirical constant.

It was realized that the exponent 0.45 for maximum density was based on reasonably equidimensional particles and thus might not yield maximum density for the flat and elongated glass particles. However, it was taken as a starting point and modified as noted later.

Six asphalt contents were chosen in 0.5 percent increments from 4.5 to 7.0 percent (total weight basis) and five specimens were made for each asphalt content. Six specimens were made each day for 5 days, with one specimen of each of the 6 asphalt contents being made each day. A hand-operated Marshall compaction hammer was used to apply 50 blows to each end of the specimen. Bulk specific gravity of each specimen was determined using ASTM Method D 2726-68 T, after which the specimens were tested for stability and flow.

Results from this first trial are given in Table 5 and shown in Figure 2. As can be seen from the graphs in Figure 2, there is no asphalt content that satisfies the Marshall design criteria suggested by The Asphalt Institute and given in Table 6. Although sta-

bility and flow are adequate at the lower asphalt contents, air content is above the minimum specified level only at 4.5 percent and voids in the mineral aggregate are below minimum allowable values at all asphalt contents between 4.5 and 6.0 percent. The data indicated that a gradation deviating from that giving maximum density was necessary.

A second series of specimens was molded using aggregate gradation No. 2 given in Table 4. This gradation had less material passing the No. 100 and No. 200 sieves. Six asphalt contents were chosen in 0.5 percent increments from 4.0 to 6.5 percent and, once

TABLE 4  
GRADATION OF AGGREGATES

Sieve Size	Gradation No. 1, Percent Passing	Gradation No. 2, Percent Passing
1/2 in.	100	100
3/8 in.	88	88
No. 4	65	67
No. 8	47	48
No. 16	35	37
No. 30	26	28
No. 50	18	18
No. 100	14	11
No. 200	9.7	6.3

TABLE 5  
MARSHALL METHOD DATA

Percent Bitumen (TWB)	Unit Weight (pcf)	Percent Air Voids	Percent Voids in Mineral Aggregate	Stability (lb)	Flow (1/100 in.)
First Trial Mix—Aggregate Gradation No. 1					
4.5	140.7	3.88	13.89	820	15.8
5.0	141.4	2.67	13.95	744	16.4
5.5	141.7	1.82	14.14	729	12.6
6.0	141.3	1.37	14.85	596	19.4
6.5	140.9	0.98	15.59	536	19.4
7.0	139.9	1.14	16.61	429	20.8
Second Trial Mix—Aggregate Gradation No. 2					
4.0	137.6	6.57	15.36	1061	7.6
4.5	137.9	5.74	15.55	736	7.4
5.0	138.7	4.50	15.57	839	7.4
5.5	139.4	3.39	15.52	767	8.0
6.0	139.5	2.62	15.93	520	10.4
6.5	139.5	1.97	16.44	492	11.0

Note: All values are averages of 5 specimens.

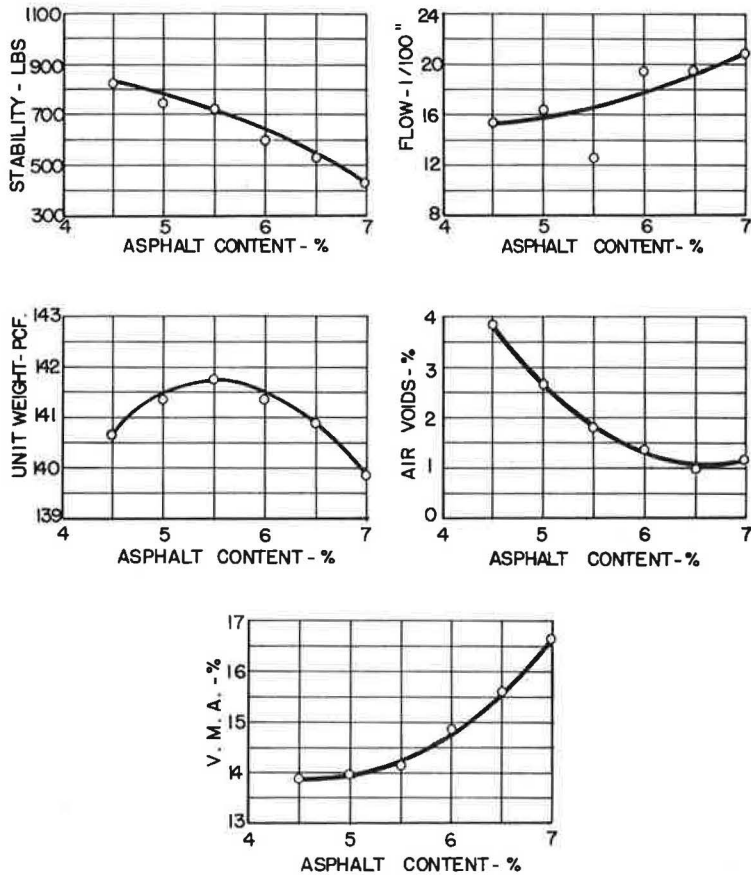


Figure 2. Marshall test property curves, first trial mix.

TABLE 6  
MARSHALL DESIGN CRITERIA<sup>a</sup>

Test Property	Minimum	Maximum
Stability	500	—
Flow	8	18
Percent air voids, surfacing	3	5
Percent voids in mineral aggregate, $\frac{1}{2}$ in. maximum size	15	—

<sup>a</sup>Recommended by The Asphalt Institute for medium traffic (50-blow compaction).

asphalt content for both trial mix series. The bitumen was removed from each of these by use of a reflux extractor with benzene as the solvent. Washed sieve analyses were conducted on the recovered aggregate (AASHTO Method T 30); the results are given in Table 7.

In order to express the degradation that occurred in terms of a single number rather than a multiplicity of percentages, a factor known as the Hudson  $\bar{A}$  was calculated (5). Hudson  $\bar{A}$  is one-hundredth of the sum of the percentages passing the ten U. S. Standard

again, 5 specimens were made for each asphalt content using 50-blow Marshall compaction.

Results from this second trial are given in Table 5 and shown in Figure 3. The reduced fines content resulted in higher voids contents and permitted the choice of an asphalt content satisfying all of the design criteria suggested by The Asphalt Institute.

#### DEGRADATION STUDIES

One specimen was chosen at random from the five specimens representing each

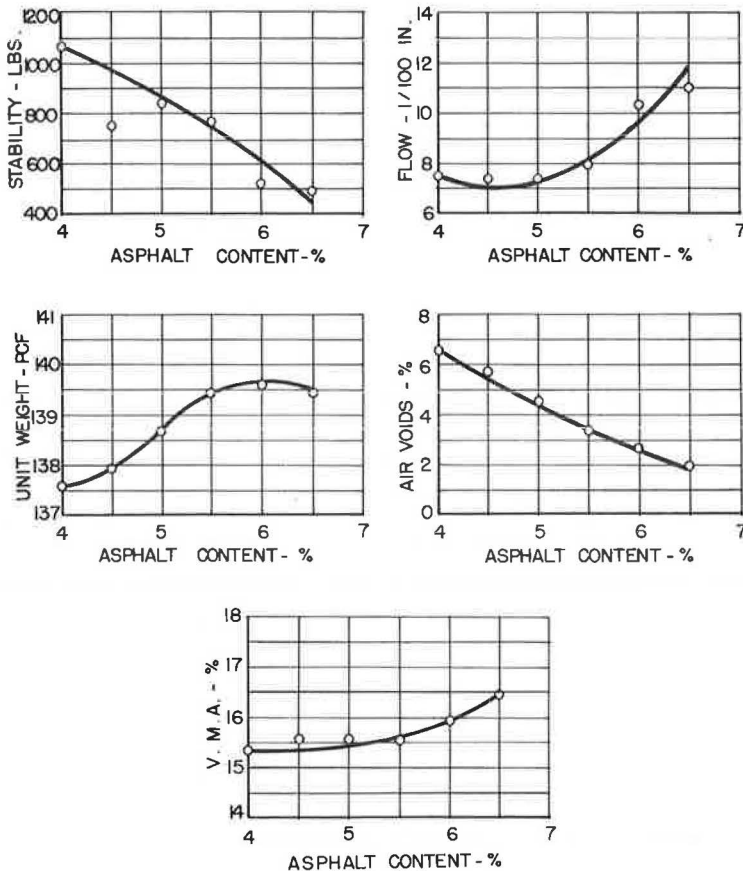


Figure 3. Marshall test property curves, second trial mix.



TABLE 7  
RESULTS OF SIEVE ANALYSES OF EXTRACTED AGGREGATES

Sieve Size	Uncompacted Percent Passing	Gradation of Extracted Aggregate, Percent Passing for Indicated Asphalt Content					
First Mix Design Series		4.5	5.0	5.5	6.0	6.5	7.0
1 1/2 in.	100	100	100	100	100	100	100
3/4 in.	88	94	94	94	93	92	93
No. 4	65	68	68	69	67	67	66
No. 8	47	49	49	49	49	48	47
No. 16	35	35	36	35	35	34	34
No. 30	26	26	26	26	27	23	25
No. 50	18	18	18	18	19	18	18
No. 100	14	14	14	14	14	13	14
No. 200	9.7	9.0	9.3	8.7	8.9	8.8	9.5
Hudson $\bar{A}$	5.03	5.13	5.14	5.14	5.13	5.04	5.07
Second Mix Design Series		4.0	4.5	5.0	5.5	6.0	6.5
1 1/2 in.	100	100	100	100	100	100	100
3/4 in.	88	95	95	94	94	93	92
No. 4	67	69	70	69	70	68	68
No. 8	48	50	50	52	51	51	50
No. 16	37	39	39	38	39	37	39
No. 30	28	28	28	28	28	27	28
No. 50	18	19	19	20	19	18	19
No. 100	11	13	12	13	13	12	12
No. 200	6.3	10.0	6.6	7.6	7.8	6.0	7.8
Hudson $\bar{A}$	5.03	5.23	5.20	5.22	5.22	5.12	5.16

sieves starting with the 1 1/2-in. sieve and including the No. 200 sieve. It has been found that, with asphaltic concrete aggregates in the usual range of  $\bar{A}$  from 4.00 to 6.00, a change of 0.50 in the value of  $\bar{A}$  would change the asphalt demand by about 1 percent by volume (5).

The results from the first trial mix indicate that little degradation of the aggregate occurred. There is an anomaly here in the percentages of material passing the No. 200 sieve. As can be seen in Table 7, the percentage of minus-200 mesh material actually decreased after the specimens had been mixed, compacted, and tested for stability. This loss of material is thought to be caused by stripping and loss of surface material on the specimens during the period in the water bath prior to testing for stability. However, it is quite apparent that very little new minus-200 mesh material was produced during mixing, testing, and compaction of these specimens and that, in general, degradation was very light as indicated by changes in Hudson  $\bar{A}$ .

Degradation was more pronounced in the second trial mix series. The Hudson  $\bar{A}$  increased by as much as 0.20 and there was a greater percentage increase in the minus-200 mesh material. The fact that more air voids were present indicates that perhaps the asphalt filler matrix was not as effective in cushioning the compactive blows, thus resulting in the formation of more fines.

#### WATER-RESISTANCE STUDIES

Static stripping tests (ASTM Method D 1664) were conducted on several combinations of glass aggregates and asphalt. Stripping was noted in the combination of glass and asphalt cement with no antistripping compound added. However, mixtures of glass and either a slow-setting cationic emulsion or an asphalt cement with a proprietary antistripping compound exhibited no stripping.

One set of 6 specimens utilizing gradation No. 2 and an asphalt content of 5.5 percent was molded to determine the percentage stability lost after immersion in water at 120 F for 4 days. However, stripping was so severe that no stability testing was possible. Tests involving the use of asphalt cements and several antistripping compounds are in progress.



## DISCUSSION OF RESULTS

The previous data have indicated that a mix satisfying the Marshall design criteria suggested by The Asphalt Institute can be designed using crushed glass aggregates and a conventional gradation. The fact that a large percentage of the particles were flat or elongated also suggests that the stability values obtained by the Marshall testing method may have been on the conservative side because of particle orientation effects. Puzinuskas (6) observed the fact that, in mixtures containing elongated or flattened aggregates, the aggregate particles tend to become axially aligned in a direction perpendicular to the direction of the compaction force. This effect was most pronounced in specimens compacted by intermittent impact-type compactive forces, such as are applied by the Marshall compactor. Specimens tested such that the compressive force was parallel to the compactive force always exhibited higher strengths than specimens tested with the force perpendicular to the compactive force. Thus, because the specimens were tested in a direction perpendicular to the direction of compaction and because a large number of flat particles were present, it is likely that the Marshall stabilities are conservative.

The data also indicate that degradation of the aggregate may be a problem in mixes deviating from a maximum density gradation. Nijboer (7) reports that a German investigator, Herrmann, has stated that the crushing of aggregate under traffic is dependent on the grading, with maximum density grading resulting in less crushing. This would appear to be the case with the two trial mixes investigated in this study, as deviations from the maximum density gradation resulted in greater increases in the minus-200 material.

The extent to which the degradation noted in the second trial mix would impair mix performance is open to question. Data obtained by Goode and Owings (8) in a laboratory and field study of degradation occurring in hot asphaltic concrete wearing course mixtures were used to obtain values for changes in Hudson  $\bar{A}$  associated with field compaction and traffic compaction. Although this parameter was not used in their paper, the data they presented permitted calculation of it for purposes of comparison. An increase in the Hudson  $\bar{A}$  value of 0.25 resulting from field compaction and an increase of 0.39 resulting from field compaction and traffic were both considered to be minor and insufficient to affect the service behavior of the pavement. These changes in Hudson  $\bar{A}$ , however, were associated with no more than a percent, more or less, increase in the minus-200 mesh material.

The use of impact-type compaction with the glass aggregate may also have resulted in more degradation than would occur under field-rolling conditions. This could be more thoroughly studied in a field installation using the glass mixture.

## CONCLUSIONS

Based on the laboratory work completed thus far, the following conclusions can be drawn:

1. Bituminous mixtures satisfying Marshall design criteria recommended by The Asphalt Institute can be designed using penetration-grade asphalts and aggregates composed entirely of crushed glass.
2. Some degradation of the glass aggregate does occur under laboratory mixing, compacting, and testing conditions. The degradation appears to increase as the original gradation deviates from a maximum density curve.
3. Severe stripping occurs when a bituminous concrete using dense-graded glass aggregates and asphalt cement with no additives is exposed to water at 120 F. Stripping is also exhibited in mixtures of asphalt cement and glass tested in the ASTM static stripping test (ASTM Method D 1664).
4. Mixtures of glass aggregates and asphalt cement treated with an antistripping compound did not strip when subjected to the static stripping test (ASTM Method D 1664). Mixtures of glass aggregates and a slow-setting cationic emulsion were also resistant to stripping in the static test.

## ANTICIPATED FURTHER RESEARCH

Further studies of the water-resistance of bituminous mixtures using glass aggregates are planned including immersion tests. A field installation consisting of patches or overlays of these mixtures is planned to assess several mixture parameters that are difficult to evaluate in the laboratory. These include workability of the mixture during placement as well as skid resistance, tire wear, and reflectivity properties of the surface.

## ACKNOWLEDGMENT

This investigation was supported by the Bureau of Solid Waste Management of the U. S. Public Health Service. The laboratory work was carried out by Charles Foster, graduate research assistant in civil engineering, at the University of Missouri—Rolla.

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## Discussion

K. O. ANDERSON, The University of Alberta—The concern expressed by the authors regarding the disposal of increasing amounts of waste glass and development of new methods for disposal of this waste component is shared by many investigators. It so happens that a similar study, in which I had a small part, has just been completed at our University (9). Although the investigation dealt with several possibilities of conversion of city refuse to useful products, including the possible recovery of metallics and heat as well as the nonmetallics, the potential use of this latter fraction of incinerated refuse as an aggregate for asphalt concrete was investigated.

In support of the authors' statement, no previous investigations of this nature were found by the discussant in the literature. As a result, a similar, although not as thorough, investigation was undertaken to evaluate the potential of this aggregate as a constituent of a hot-mix asphalt paving mixture. Standard mix design procedures, following ASTM Method D 1559 but modified slightly to include a measure of loss in stability resulting from the action of water, were also used.

The nonmetallic fraction of the incinerated refuse accounting for approximately 65 percent of the total was crushed and separated into seven fractions and recombined to meet requirements for Type IVb Asphalt Institute gradation. A 200-300 penetration grade of asphalt cement was used without any additives. A mechanical compactor was used to impart 50 blows per face at the recommended compacting temperature based on viscosity. Initial stability and flow values were obtained on specimens brought to

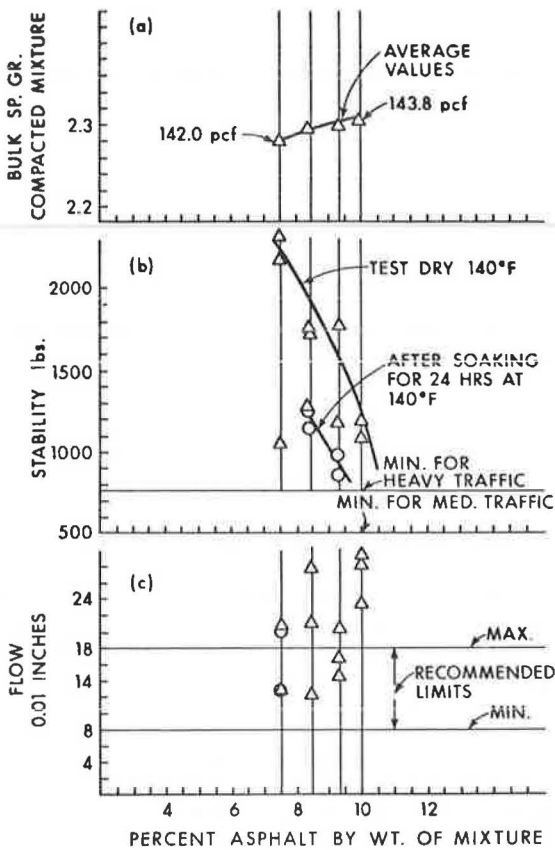


Figure 4. Results of tests on asphalt paving mixture using nonmetallic incinerator refuse as aggregate.

140 F in an air bath, and comparative values were obtained after soaking in water at 140 F for 24 hours. Results obtained are shown in Figure 4.

Although insufficient tests were performed to establish optimum conditions for this mix, it was apparent that surprisingly high stability values could be attained, even after the period of immersion. Even though the retained stability values were 69 and 59 percent for the 9 and 10 percent asphalt series respectively and below a generally accepted minimum of 75 percent retained stability, the absolute values were still above the minimum stability requirements for medium traffic as suggested by The Asphalt Institute. The flow values were very erratic; however, some were within the prescribed limits.

On the basis of these preliminary tests, we were able to state that it would appear possible to use this material in an asphalt paving mixture. This would be in support of the first conclusion by the authors. The less severe stripping in our tests, in contrast to the third conclusion of the paper, may be due in part to the fact that the incinerated refuse contained minor amounts of porcelain, rock, some metallics, and fly ash that may have increased the resistance to stripping of our mixture. It will be interesting to hear of further studies on this very timely topic.

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WARD R. MALISCH, DELBERT E. DAY, and BOBBY G. WIXSON, Closure—The authors appreciate the supporting comments and the additional information provided by the discussant. The possibility that incinerator residue can be used with asphalt to produce a reasonably water-resistant mixture without the use of antistripping agents is further indication that this may be a feasible means for reutilization of solid waste materials. The higher stabilities that were obtained by Anderson would also seem to indicate a difference in surface texture or shape between the crushed bottles used in our investigation and the incinerator residue. Continuing research should further clarify this point.

# Construction Specifications for Volcanic Cinders Used as Road-Surfacing Aggregate

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The purpose of this study was to determine parameters that influence the performance of volcanic cinders when used as a wearing surface for unpaved forest access roads. The steady depletion of good, high-type aggregate sources has brought about renewed interest in other aggregate types, making this study particularly timely. A literature review, correspondence, and personal interviews were conducted to determine existing use and performance of cinders. A road rating system was developed, similar to the AASHO Road Test method, to determine which were "good roads" and which were "poor roads" based on a 0 to 5 numerical rating scale. Thirty of the rated roads and the corresponding cinder pits were sampled and the material was tested in the laboratory. The physical properties of the cinders were then statistically correlated with the road rating. It was found that density, gradation, durability, and plasticity were the most significant independent variables. Based on these results, specifications for untreated cinder surface courses have been developed. These include (a) a minimum of 100 percent compaction relative to AASHO T 99; (b) gradation limits of 100 percent passing the 1-in. sieve, 80 to 95 percent passing the  $\frac{3}{4}$ -in., 35 to 60 percent passing the No. 4, 22 to 45 percent passing the No. 10, 8 to 25 percent passing the No. 40, and 3 to 12 percent passing the No. 200; (c) a plastic index value between 2 and 10; and (d) a maximum Los Angeles abrasion value of 50 prior to processing. In most cases this will require crushing of the harder (purple, gray, and black) cinders.

•VOLCANIC CINDERS occur in many areas of the world and are particularly common in central Oregon, which is the location of this study. The Forest Service of the U. S. Department of Agriculture builds many miles of untreated aggregate-surfaced roads in the states of Oregon and Washington. In the central Oregon area, the use of volcanic cinders to surface those roads is very common. The performance of cinder-surfaced roads has varied, with both good and poor results. The general trend among many highway agencies is to avoid their use. However, the steady depletion of good, high-type aggregate sources has brought about renewed interest in other aggregate types, including volcanic cinders. Present use of volcanic cinders is primarily for low-class roads, either untreated or with a light asphaltic surface treatment.

## PURPOSE OF STUDY

The purpose of the study was to determine the parameters that influence the performance of volcanic cinders when used as a wearing surface and base course for forest development roads. The approach to this problem suggested an evaluation of the

performance of existing untreated cinder roads to determine which were "good roads" and which were "poor roads" based on a numerical rating scale. This, in turn, was related to the physical properties of the cinders in the roadway and from the borrow pit. Included in the study was a survey to determine current practice and associated problems involved in the use of cinders as road aggregates. These relationships, together with other field observations, were used to recommend specifications for selection of borrow material and for techniques of construction quality control.

This paper is concerned with the relationship between cinder properties and road-surfacing performance. The details of the entire study, giving the background of the road-rating system and the results of the survey to determine current use, are presented in a U. S. Forest Service report (1).

## BACKGROUND INFORMATION

Cinders, as referred to in this report, are a pyroclastic material associated with volcanic activity (2, 3). Fragmented material of this type is generally classified according to size. Pieces larger than approximately 10 in. (256 mm) in diameter are called blocks or breccia if angular and ejected in a solid state and are called bombs if ejected in a plastic state, which produces a roundish or ellipsoidal shape with twisted ends. Pieces between 10 in. and  $\frac{1}{16}$  in. (4 mm) in diameter are called lapilli or cinders. Particles finer than approximately  $\frac{1}{16}$  in. in diameter are called ash or dust (4).

A cinder, unlike most rocks, does not have a set mineral or chemical composition because it is mainly a textural classification of rock. It may be either acidic (light-colored matter consisting principally of quartz and feldspars) or basic (dark-colored matter consisting principally of ferromagnesian minerals with no quartz). Color reflects the conditions prevalent during formation (i.e., temperature or oxygen available) and weathering. Oxygen-deficient cinders are darker in color, whereas red or brownish cinders generally indicate weathering and are located in the upper zone of a deposit. Minor accessory minerals, such as iron, may also contribute to the coloring of the cinder.

Scoria is a term used to describe any volcanic ejecta that is rough, sharp, and vesicular, either pyroclastic material or the upper surface of some lava flows. It is commonly black or reddish and principally basic in composition. The term is often used to include cinders.

Cinders are located throughout the world. The main areas in the United States are central Oregon east of the Cascades, northern and eastern California, Arizona, New Mexico, and Hawaii. Notable deposits are found in New Zealand, Japan, Turkey, Mexico, Central America, and the Caribbean. In other countries the terms scoria, scoriae, volcanic agglomerate, or grits are used more often than cinders, especially by British Road Research Laboratory reports (5, 6, 7).

## Current Practice and Experience

According to an earlier publication (8), almost all agencies will avoid the use of cinders if other sources of mineral aggregate are available. When used, cinders are applied as surfacing material primarily on unpaved roads, such as logging, forest access, and low-traffic roads. On higher type roads cinders are used as subbase and base material and for asphalt-stabilized surfacing.

The best results with pit-run cinders have been with the softer types (generally the red-colored types; average Los Angeles abrasion in the 40s). The harder types (purple, gray, and black; average Los Angeles abrasion in the 30s) lack fines, and thus are unstable (Fig. 1). When crushed, the harder types perform more satisfactorily, because the necessary fines are obtained and thus the surface better withstands the abrasive effects of traffic. In addition, the rough riding quality resulting from the pit-run over-sized material is eliminated.

Grid rollers are popularly used for breaking the larger pieces and for compaction. The effectiveness of this type of roller for compaction is questionable. Steel wheel, rubber-tired, and vibration rollers appear to achieve better results, as does compaction from logging and other heavy truck traffic.





Figure 1. Sycan Road, North of Bly, Oregon: Note contrast between washboard surface using purple cinders (foreground) and smooth surface using red cinders (background).

With increasing traffic, new road construction and pavement thickness upgrading, and the lack and depletion of good mineral aggregate sources, the use of cinders is being investigated in more detail. Many agencies feel that cinders can be used effectively with proper gradation and other quality controls. Asphalt treatment will probably give the best road surface and the most economical maintenance over an extended period.

#### CORRELATION OF CINDER PROPERTIES WITH SURFACING PERFORMANCE

##### Development of the Rating System

A road-rating system was developed similar to the AASHO Road Test methods. A major differentiation was that for this study the roads were rated over a period of time; thus, the roads were given a performance index (PI) rating rather than a serviceability rating. Forty-seven roads were rated on a scale from 0 to 5 with performance increasing with increasing rating values (Figs. 2 and 3). This rating was performed by people familiar with the long-term serviceability of the road. After the rating was obtained, it was correlated with maintenance frequency, rutting, watering, speed, and traffic volume by means of a multiple linear regression computer program. The resulting regression equation had a multiple correlation coefficient of 0.812. This equation was then used to determine the performance index of the various roads, which was, in essence, the performance rating with the individual rater's bias removed. Complete details of this rating technique can be obtained by referring to the U. S. Forest Service report (1).

##### Road- and Pit-Sampling Program

Once the road-rating system was established using the PI, the next step was to select typical examples of roads within each rating group and to sample the surface material. In all, 30 roads and their corresponding borrow-pit sources were sampled, including 15 in the Winema National Forest, and 5 each in the Fremont, Rogue River, and Deschutes National Forests. The general location of the sample sites is shown in Figure 4.

The purpose in sampling these roads and pits was to determine physical properties that could be identified with roads of high performance (PI) and those associated with



Figure 2. Kirk Road, Northeast of Chiloquin, Oregon: Rough surface contains black, uniformly graded cinders (PI = 2.1).

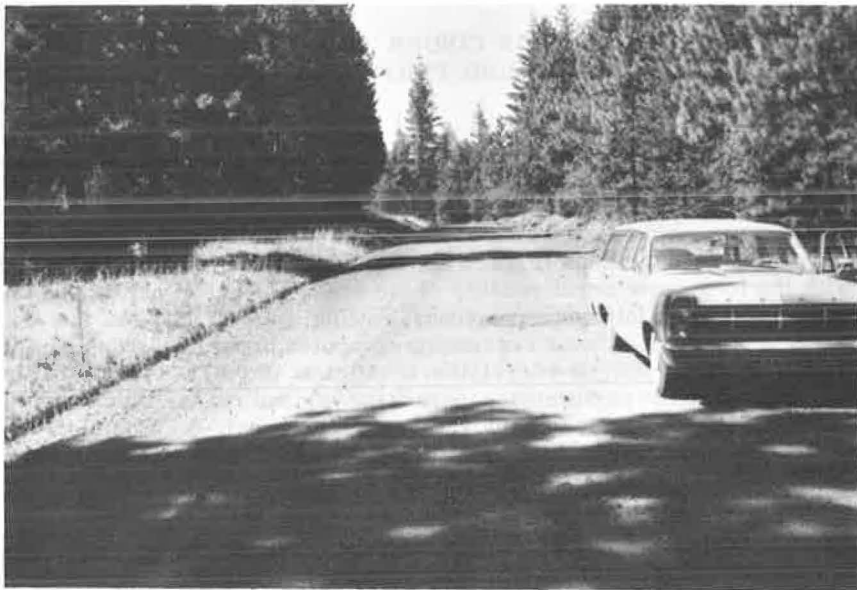


Figure 3. Twincheria Road, East of Butte Falls, Oregon: Cinders cemented with iron oxide produce a hard, compact riding surface (PI = 3.1).

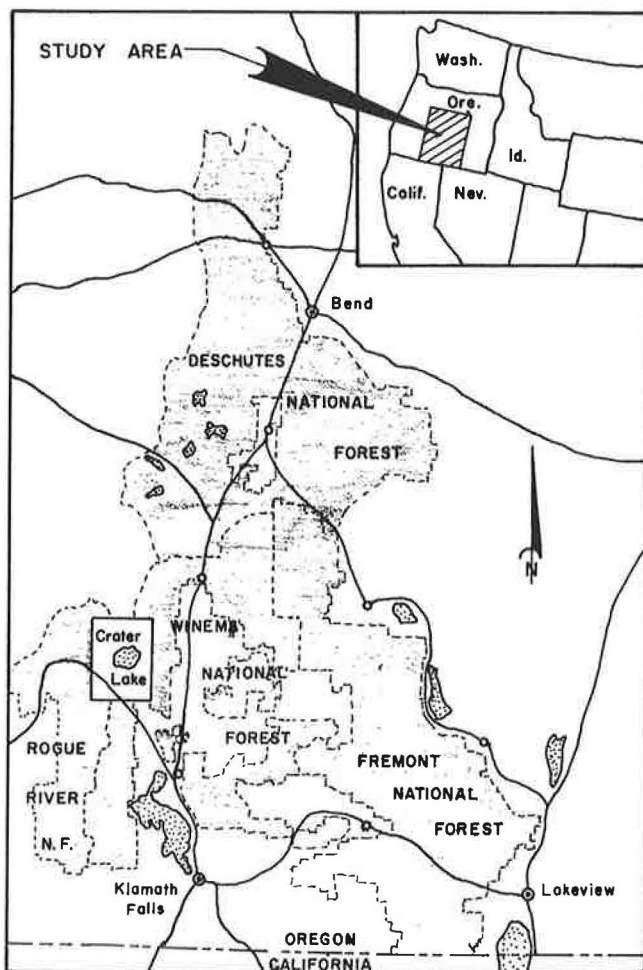


Figure 4. Study location map.

low performance, or more specifically the correlation between performance and physical property. All roads were constructed of pit-run material.

The testing program consisted of two parts, field sampling and testing, and laboratory testing. The field sampling and testing portion involved first selecting a straight section of the rated road and obtaining approximately 150 lb of material (2 sample sacks). This was obtained from a trench dug at least halfway across the road and through the majority of the surface course (approximately 4 to 6 in. deep). In addition, the following field tests were performed: (a) in-place density using the Washington densometer; (b) in-place moisture content using the Speedy moisture tester; (c) color identification; and (d) penetration test of surface and base material (later discontinued because there appeared to be no correlation).

The pit site was located next and a 75-lb sample (1 sample sack) was obtained from the approximate area in the pit where the road sample was obtained during construction. Obtaining a related sample was sometimes difficult because of the mixing of pit material and subsequent use of the pit for other construction projects.

The samples were sent to the Forest Service Regional Office in Portland, and in turn sent to the Federal Highway Administration laboratory in Vancouver, Washington, or to the Oregon State Highway Department (OSHD) laboratory in Salem (Table 1).



TABLE 1  
TESTS PERFORMED ON ROAD SAMPLES

Test Description	Test Method	Tests Performed on	
		Road Samples	Pit Samples
Los Angeles abrasion	AASHTO T 96	X	X
Specific gravity of fines	California 208B	X	
Unit weight, dry-rodded		X <sup>a</sup>	
Unit weight, loose		X <sup>a</sup>	X <sup>a</sup>
Plasticity index	AASHTO T 99	X	X
Liquid limit	AASHTO T 89	X	X
Sand equivalent	AASHTO T 176	X	
Oregon air degradation	OSHD	X	X <sup>a</sup>
Moisture-density relationship	AASHTO T 99 D	X	
Gradation (dry sieve analysis)	AASHTO T 27	X	X

<sup>a</sup>These determinations were made only on samples sent to the Oregon State Highway Department (OSHD).

Splitting of the work was necessary because of the volume of testing involved and the time required. Some additional testing was also performed at the Forest Service Engineering Materials Laboratory in Portland, Oregon.

In addition to the testing, the following calculations were performed: (a) coefficient of uniformity ( $C_u$ ) for both road and pit gradation; (b) coefficient of curvature ( $C_c$ ) for both road and pit gradation; and (c) relative compaction of road sample (as compared to maximum density). Several points should be made in regard to the test results:

1. Los Angeles abrasion test—The actual values for the Los Angeles abrasion appear to be very good (low values) and this is due in part to the volume of material used in running the test. The standard charge is 5,000 grams of material. With the low bulk specific gravity of cinders, a greater volume of material is used than would be normally utilized (9). Because this study considers only cinder ag-

gregate, relative values between various cinder samples should be acceptable for use in the analysis.

2. Specific gravity test—Unfortunately this test was run in a Le Châtelier flask, (Calif. 208B); thus, only material passing the No. 4 sieve was used and the apparent specific gravity was determined. As a result, the values were very uniform, whereas values for the coarse portion would probably have been more meaningful.

3. Plasticity index and liquid limit—In most cases the cinders had very few plastic fines, thus giving no plastic limit. However, the liquid limit could be determined for most samples (often only by one point and extrapolation) and was thus reported because it appeared to give good correlation.

### Correlation With Performance Index

The results of the field sampling and laboratory testing were compared with the performance index (road rating) by means of the multiple regression program. Nineteen independent variables from road tests and 12 independent variables from pit tests were considered. These variables are listed in table 2 together with their average values, standard deviations, and correlations with the performance index.

According to the regression program, the following are the most significant variables (with correlation coefficients above 0.3) in decreasing order of importance. The values for the percent passing the  $1\frac{1}{2}$ - and  $\frac{3}{4}$ -in. sieves are results from pit source samples.

Variable	Correlation With Performance Index
Relative compaction	0.395
Plasticity index	0.392
Percent passing No. 4 sieve	-0.341
Percent passing $1\frac{1}{2}$ -in. sieve	0.328
Percent passing No. 10 sieve	-0.325
Coefficient of curvature	0.323
Oregon air degradation—H	0.319
Percent passing No. 40 sieve	-0.312
Percent passing $\frac{3}{4}$ -in. sieve	0.310

TABLE 2  
CORRELATION OF ROAD AND PIT TEST VARIABLES

Variable	Mean	Standard Deviation	Correlation With Performance Index
Road Test Variables			
Sand equivalent	49.4	24.0	-0.130
Los Angeles abrasion, percent	43.8	10.4	-0.099
Maximum density, pcf	102.5	7.6	-0.058
Relative compaction, percent	102.4	5.7	0.395
Specific gravity	2.72	0.12	-0.154
Plasticity index	0.1	0.4	0.392
Liquid limit	23.8	4.2	0.196
Oregon air degradation, sediment height, in.	3.5	4.7	0.319
Oregon air degradation, percent passing No. 20 sieve	26.4	9.8	-0.024
Color (red = 2, black = 1)	1.8	0.4	-0.183
Coefficient of uniformity	52.6	31.4	0.269
Coefficient of curvature	0.72	0.56	0.323
Gradation, percent passing sieves			
1 1/2 in.	96.1	5.6	-0.119
3/4 in.	88.9	6.9	-0.117
3/8 in.	76.2	7.4	-0.213
No. 4	61.4	8.6	-0.341
No. 10	50.1	9.5	-0.325
No. 40	31.1	8.0	-0.312
No. 200	9.6	2.8	-0.243
Pit Source Variables			
Los Angeles abrasion, percent	46.7	10.6	-0.029
Liquid limit	28.5	3.3	-0.082
Unit weight, loose, pcf	67.1	7.5	-0.073
Coefficient of uniformity	38.1	22.6	-0.071
Coefficient of curvature	1.60	1.21	-0.127
Gradation, percent passing sieves			
1 1/2 in.	96.1	2.8	0.328
3/4 in.	86.0	6.5	0.310
3/8 in.	69.0	9.8	0.286
No. 4	50.5	12.8	0.247
No. 10	37.1	13.4	0.252
No. 40	18.5	8.4	0.090
No. 200	4.6	2.5	0.095

The multiple regression equation is as follows:

$$PI = -5.65 + 0.06(P) + 0.05(RC) + 0.08(OA-H) - 0.04(OA-20) + 0.06(PP1.5) - 0.07(PP4) - 0.24(Color) + 0.09(PP10) - 0.02(PUW) + 0.30(CC)$$

where

- PI = performance index;
- P = plasticity index;
- RC = relative compaction;
- OA-H = Oregon air degradation, sediment height;
- OA-20 = Oregon air degradation, percent passing No. 20 sieve;
- PP1.5 = percent passing 1 1/2-in. sieve from pit;
- PP4 = percent passing No. 4 sieve from pit;
- Color = cinder color (red = 2, black = 1);
- PP10 = percent passing No. 10 sieve from pit;
- PUW = loose unit weight of material from pit; and
- CC = coefficient of curvature from road.

This equation has a multiple correlation coefficient of 0.854. Individual equations were also determined for each of the 9 most significant variables. The equations and the individual correlation coefficients indicate the relationship between the physical test property and the performance index (rating). Unfortunately, 24 of the 32 roads had a performance index within the relatively narrow range of 2.1 to 3.5, with only 2 roads rated below this range. Thus, the regression analysis gave the greatest weight to this range, with the result that most of the regression lines have very steep slopes.

Several important trends were noted when comparing the test results with the performance index. They are summarized in the following:

1. Compaction—In the tests of road samples, the relative compaction is very important, whereas the maximum density is not.

2. Gradation—In the tests of road samples, the coefficient of curvature varies directly with the performance index (i.e., gradations with a concave-shaped curve on a semilog graph are associated with the higher performance index). This relationship is also reinforced by the trend showing less material passing the No. 4, No. 10, and No. 40 sieves with a higher performance index; the value for the other sieves appears not to be significant. In the tests of pit samples, the increased percentages passing the 1½-in. and ¾-in. sieves is desirable (i.e., less large-size material).

3. Plasticity—In the road sample tests, the significant characteristics of the fine material appear to be increased plasticity index, liquid limit, and sediment height in the Oregon air degradation test, and decreased sand equivalent with increasing performance index (i.e., cohesive binder is desirable).

4. Abrasion—In the road sample tests, the Los Angeles abrasion value varies inversely with the performance index (i.e., harder material with less abrasion is desirable). In the pit sample tests the decreased Los Angeles abrasion value is desirable.

#### Recommended Specifications and Field Quality Controls

Based on the physical test results, the cinder characteristics that appear to be significant are compaction, gradation, plasticity, and resistance to abrasion. Using these characteristics, the following specifications are recommended for the construction of cinder wearing surfaces.

**Compaction**—In all cases the final constructed surface should be compacted to at least 100 percent of standard compaction (AASHTO T 99). This should extend to a depth of 6 in. below the surface. In most cases, it is recommended that a vibratory compactor be utilized, or as an alternate a pneumatic roller. Sheepfoot and grid rollers should be avoided. Steel-wheel rollers can be used for finish or proof rolling.

**Gradation**—The maximum-sized particle should be 1 in. in diameter in the finished surface. In certain cases, this size could be increased to 1½ in. at the pit source, as some degradation will take place in the construction process. The following in-place road gradation is recommended (based on dry-sieving similar to AASHTO T 27, with a maximum shaking time of 5 minutes):

<u>Sieve Size</u>	<u>Percent Passing</u>
1 in.	100
¾ in.	80 to 95
No. 4	35 to 60
No. 10	22 to 45
No. 40	8 to 25
No. 200	3 to 12

These limits will provide a dense gradation.

This gradation is similar to AASHTO M 147-55, grading C, and U. S. Forest Service Item 151, grading B, Item R151, grading C, and Item R150, grading C-1. The gradation recommended in this report differs somewhat in that it has a smaller percentage passing in the No. 4 to No. 40 sieve range to allow for subsequent degradation of the cinders. Local practice may indicate that the standard specifications listed previously are adequate.

It is realized that in-place road gradations are often difficult to estimate on the basis of pit-sampling. The best technique for sampling cinder pits is to obtain as representative a sample as possible (i.e., for the portion of the pit to be used, or a blended sample). Based on results from this study, the pit gradation should have approximately 5 percent less passing a given sieve (below the  $\frac{3}{4}$ -in. size) than is desired for the finished product on the road. This allows for degradation in transporting and compacting the cinders. This value should be approximately 10 percent for the softer red and brown cinders.

The recommended gradation requirements could be met by selected blending and scalping at the pit source. However, in some cases it may be necessary to run the material through a primary crusher with some selective screening.

Control of gradation is felt to be extremely important because too much large material (above 1-in. size) causes a rough riding surface, and too much fine material will cause excessive and early washboarding and rutting.

Atterberg Limits—The material passing the No. 40 sieve should have the following characteristics: The liquid limit should be 35 at the maximum, and the plasticity index should range from 2 to 10.

Based on the trends illustrated, sand equivalent test results ranging from 50 to 30 would give an indication of the required plasticity. This value should not, however, be used as a specification requirement because it measures somewhat different properties of the fine material.

This requirement of plasticity is felt necessary to replace evaporated surface moisture through capillarity. In most cases, cinders do not produce this plasticity, and thus the addition of plastic binder is necessary. This binder may be available as a cap of weathered cinders over the cone, or can be generated in some cases by watering, mixing, and compacting the softer brown and red cinders. A pulvemixer attached to a small tractor would perform an effective job of blending the cinders and plastic borrow material. In some pits, the presence of an iron oxide binder will give a satisfactory binding action in the road. If this is the case, the plasticity requirements could be waived.

If immediate paving of the cinder surface with an impervious mat is anticipated, then the plastic material should be avoided. If paving is contemplated at a later date, then lime, asphalt, cement, or other additives should be considered to reduce or eliminate the plastic characteristics at this time.

Resistance to Abrasion—Maximum pit Los Angeles abrasion of 50 should be used to prevent excessive degradation of the material in the road surface, because excessive fines will cause early washboarding and rutting. With pit-run material, this degradation was necessary to generate sufficient fines for binder. However, with the recommended limits on gradation, this will no longer be necessary or desirable.

## CONCLUSION

In most cases, the harder cinders (purple-, gray-, and black-colored ones) should have less degradation and provide a better riding surface over a longer period of time. However, this type of cinder usually lacks the necessary fines, and thus some crushing is required to meet the recommended gradation limits. The softer cinders (red- and brown-colored ones) sometimes have excessive degradation; thus, care must be exercised in selecting the necessary gradation. The Los Angeles abrasion requirement will eliminate the use of most of the softer types. In some pits, where several cinder colors are available, blending of the harder and softer types may produce the necessary gradation and plasticity to provide an adequate riding surface.

## SUMMARY

The foregoing analysis shows that cinders behave much as any other granular material when used as an untreated road surface. The study revealed that several modifications were desirable to standard specifications to better fit the properties of cinder aggregates. The test data indicate that a Los Angeles abrasion value of 50 would be more appropriate than the usual limit of 40. The mean of all Los Angeles abrasion values is a 46.7 percent loss, with a standard deviation of 10.6 when sampled from the pit. Therefore, a maximum allowable limit of 40 is too restrictive. The test data show



that both the plasticity index and the gradation are very important for good performance. The gradation should ideally be somewhat on the coarse side of the gradation band to allow for subsequent degradation under traffic. Relative compaction was also found to be very important, which further emphasizes that cinders behave much as any other granular material used as a road aggregate.

Reference to Table 2 shows a minor correlation (low value) between Los Angeles abrasion and performance index (PI) and a positive, strong correlation between the Oregon air degradation sediment height (H) value and PI. However, it is recommended that the Los Angeles abrasion test be used as a control test rather than the Oregon air degradation test. This is because high-quality aggregate gives low Oregon air H values. Because this study showed a positive correlation between H and PI, the statistics indicate that the poorer quality material yields higher PI values. This must, however, be considered in light of the fact that, for all the roads studied, the surfacing was placed with almost no gradation control and the surfacing was almost always lacking in fines. Therefore, when the material had a high H value, the cinders tended to break down under watering and traffic to produce the needed fines. Thus, there was a strong correlation between H value and PI. The recommendations in this report are to control the gradation before placing the material; consequently, the desirability of having a high H value is no longer valid. There was a negative correlation between the Los Angeles abrasion and PI; therefore, a specification for Los Angeles abrasion is recommended. If a similar study were performed on roads where cinders were applied under a controlled gradation specification, it is felt by the authors that the Los Angeles abrasion test would show a much stronger negative correlation. For this reason, and because of the obvious need to specify a material that will tend to hold its gradation, the Los Angeles abrasion test is recommended. The maximum allowable limit was based on the mean and the standard deviation of the test results.

It is believed that the specifications recommended are reasonable, based on test results of typical cinders in central Oregon and the proven, accepted practice for aggregate surfacing control. The typical specifications when inserted into the regression equation yield a performance index over 5.0, which is an indication that the criteria are consistent with the test results.

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# Lightweight Concrete Aggregate From Sintered Fly Ash

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Lightweight concrete aggregate is being produced commercially from pulverized coal fly ash by several plants. The products are of good quality, although they vary somewhat with respect to shape and size. The type of fly ash used in the manufacturing process is generally that produced from bituminous coal. The characteristics of fly ash that are required in the forming and sintering process are described. A description of the physical properties of the fly ash aggregate is presented. The evaluation of these aggregates by the use of ASTM methods gives a good indication of the performance of the various materials in structural concrete. Comparisons are provided between other types of lightweight aggregate concrete and, in all cases, the fly ash concrete shows a favorable range of properties. Concrete strengths in excess of 6,000 psi (420 kg/cm<sup>2</sup>) can be produced with fly ash aggregate. Of significance in making comparisons is the high rate of absorption of the aggregate, the residual pozzolanic activity, and the relatively low cement factors required for lean concrete. Information is presented on typical mix designs for use in structural concrete.

•THE PRODUCTION of lightweight aggregate from fly ash has received the attention of investigators throughout the world (1,2). Although a number of early attempts to produce a commercial product proved unsuccessful, recent developments give every indication that the manufacture of fly ash aggregate is now possible. Furthermore, the product quality is quite acceptable for use in structural concrete, for insulation-type concrete, and as an aggregate for masonry units.

Fly ash serves as an exceptionally good raw material because it is available in large quantities in geographic areas where lightweight aggregates are in demand. The commercial methods in use today produce aggregate by first preparing an unfired pellet or granule of the fly ash followed by firing on some type of sinter strand. Because fly ash usually contains some unburned carbon, there is an opportunity to utilize the available heat generated by burning of residual combustible material during processing. Fly ash aggregates, therefore, are properly classified as "sintered" products rather than "bloated" materials, such as those produced by heating clay or shale in rotary kilns.

Historically, coal ash has often found application as an aggregate. Originally this was in the form of "cinders" from traveling grates or stoker-fed coal burners. These systems have now been largely replaced by pulverized-coal burners and the resulting fly ash is collected in a very finely divided condition in various types of dust-collecting systems, such as cyclone collectors or electrostatic precipitators.

It is the purpose of this paper to describe the requirements for fly ash as used in the sintering processes and the properties of the fly ash aggregates that are produced. Although it is not intended to present the techniques involved in the production of aggregates, some information is included relating to the effect of the manufacturing process on the characteristics of the aggregate.

TABLE 1  
TYPICAL FLY ASH ANALYSES FOR VARIOUS TYPES OF COAL

Sample Source	LOI	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>
Bituminous							
Illinois	2	44	19	19	8	1	4
Kentucky	2	42	20	25	4	<1	2
Ohio	10	41	23	21	1	1	<1
West Virginia	6	41	24	21	4	<1	<1
Pennsylvania							
Source 1	10	47	26	11	2	1	<1
Source 2	5	38	24	26	4	1	1
Source 3	6	40	24	20	6	1	2
Source 4	20	40	23	12	2	<1	<1
England	2	48	26	10	4	2	1
Poland	4	50	26	12	5	2	1
West Germany	3	44	27	12	4	3	<1
Lignitic							
Minnesota	1	23	12	14	32	10	3
Montana	<1	28	10	11	30	17	1
North Dakota	<1	31	13	11	24	11	4
North Dakota	4	23	16	8	36	9	4
Canada	1	39	21	7	19	5	2
Czechoslovakia	3	58	11	11	15	2	2
France	<1	58	9	22	6	2	2
Greece	2	38	18	9	28	2	3
East Germany	<1	39	7	21	21	2	10
Poland	2	34	8	6	38	4	9

## RAW MATERIALS

The types of fly ash available cover a broad spectrum of chemical and physical properties. Table 1 gives some typical analyses of fly ashes from various parts of the world. There are several factors that affect the quality of ash as used in the manufacture of aggregate. Of prime importance is the type of coal used; thus, ash produced from bituminous coal is usually quite low in calcium and magnesium oxides, whereas ash produced from lignite coal is usually high in these two constituents. The presence of excessive amounts of free calcium oxide in a fired aggregate can have deleterious effects; among these is hydration of the oxide, resulting in the formation of "pits" or "pops" in the hardened concrete.

A second factor affecting the quality of the fly ash is the ash content of the coal. The majority of coals used in the United States is relatively low in ash, usually approximately 10 percent. The coals in Europe are frequently quite high in ash content (up to about 45 percent). European practice, therefore, requires that the coal be burned more thoroughly to recover as much fuel as possible. The resulting fly ash is low in residual carbon—in many instances less than 1 percent. Carbon contents in the United States are rarely this low and it is not uncommon for the carbon content of the ash to be in excess of 10 percent. It is essential that fly ash contain not less than about 4 percent and preferably not more than 8 percent carbon to be an acceptable material for aggregate production.

A third factor affecting the fly ash quality is the type of ash collection systems in use. This can directly influence the fineness of the ash. Fineness varies not only between different coal-burning operations but also within a given operation. For example, in a typical collection system used in this country there may be as many as 20 hoppers removing ash from the furnace. The location of these hoppers in relation to the gas flow results in substantial differences in the particle size of the ash.

The variation of properties of the ash influences to a considerable degree the quality control of the sinter strand operations. Thus, if a fly ash changes in its fuel content (residual carbon) from a low to a high value as it is being fed into the firing process, serious difficulties can be experienced in controlling the operation. Variation in iron oxide content is of considerable importance because this ingredient serves as a primary

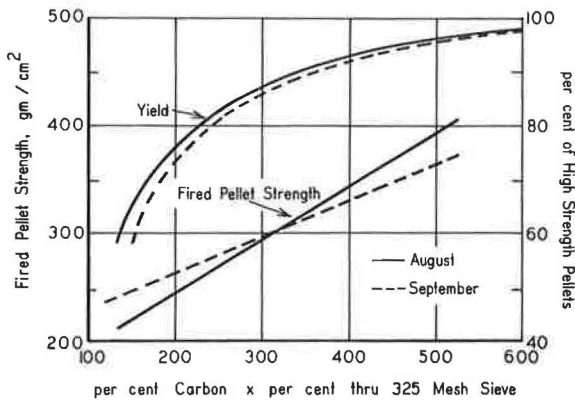


Figure 1. Effect of carbon content and sieve fineness on pellet strength and percentage of high-strength pellets.

Figure 1 shows the effect of carbon and No. 325 mesh sieve fineness on both the percentage of high-strength pellets and the fired pellet strength on a large number of samples (at a relatively constant  $\text{Fe}_2\text{O}_3$  content) taken at different periods of time from one power plant. Although carbon content and sieve fineness do not give a high degree of correlation individually, the product of the percent of carbon times the percent through the No. 325 mesh sieve does, as shown in Figure 1. Based on studies such as these, limits can be established to provide adequate quality control of the raw fly ash for use in the sintering processes.

The interrelationship between fineness and carbon (also  $\text{Fe}_2\text{O}_3$  content) can be useful in arriving at the selection of fly ash material at the power station. Although fly ash from both mechanical and electrostatic collectors can be used, there are situations that require the rejection of a portion of the fly ash that does not conform to the general requirements of the lightweight aggregate process.

In addition to the control of properties of the raw material, an important factor in the preparation of the aggregate is the water content used in the formation of the green pellets prior to firing. The water is necessary not only to develop adequate physical strength of the pellet but also to control the porosity or absorption of the fired aggregate.

#### SHAPE AND SIZE OF FLY ASH AGGREGATE

Although there are a few exceptions, most fly ash aggregates develop the ultimate shape and volume of the individual particles at the time the material is formed in the unfired (green) state. Several devices are currently used to form the unfired pellets or granules. Pelletizing drums or pans are used in a few plants. These particular devices form spherically shaped particles usually of a very uniform diameter. In the process developed by the Corson Company, an extrusion principle is used that results in particles of varying shapes and sizes, depending on the selection of the forming equipment. Figures 2, 3, and 4 show typical shapes produced by the different forming processes.

One significant factor resulting from the choice of the forming method is the final density that is achieved in the aggregate. Furthermore, this density also contributes, to a substantial degree, to the absorption of the aggregate. The commercial operations are all engineered to produce a product that retains the discrete shape of the unfired pellet without formation of large clinkers, which are often produced from other types of raw materials.

From the standpoint of producing aggregate for structural concrete, the range of sizes of the pellets are set to meet the basic requirements of the ASTM specification for concrete aggregates. In order to get the proper gradation, usually several methods

flux and substantially influences the operation of the plant process as well as the product quality.

As a result of an intensive study made in the author's laboratory, it has been found that the most important requirements for suitable fly ash are the carbon content (or loss on ignition), the fineness (as measured with a No. 325 mesh sieve), and the iron oxide content. It has also been found from statistical studies that these individual characteristics do not reliably predict the resultant aggregate properties, but that when these characteristics are used as multiple factors they give significant relationships between the fly ash and the properties of the final product.



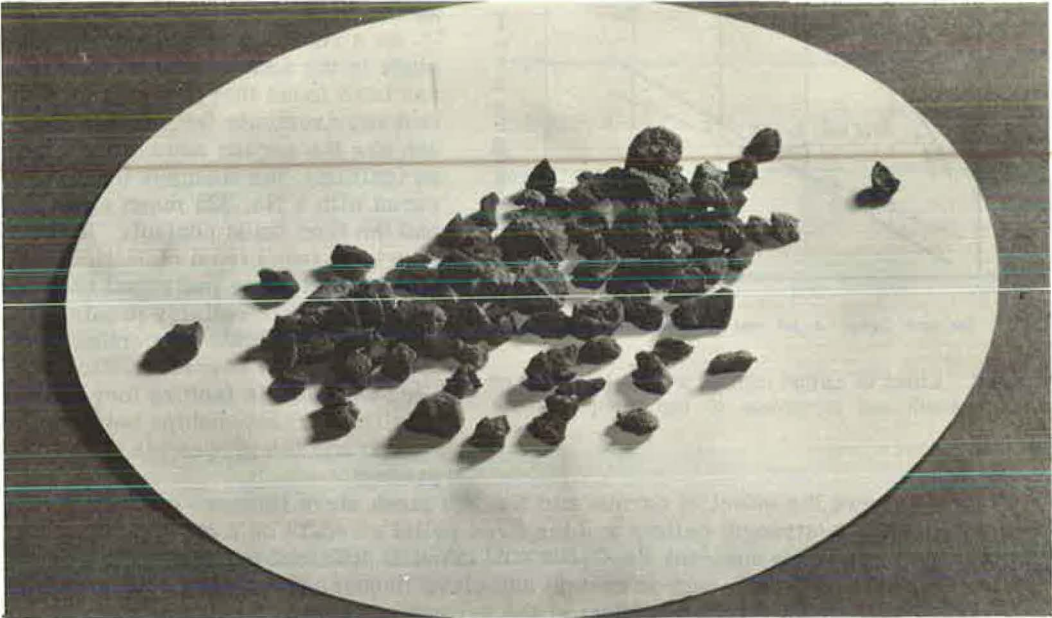


Figure 2. Extruded fly ash aggregate (crushed and graded).

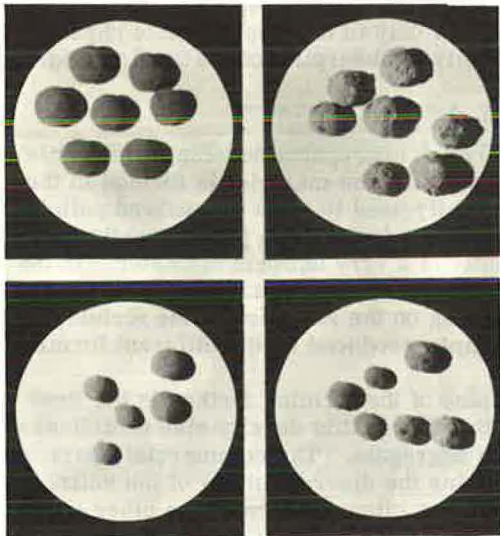


Figure 3. Unfired and fired fly ash aggregate made by pelletizing process.

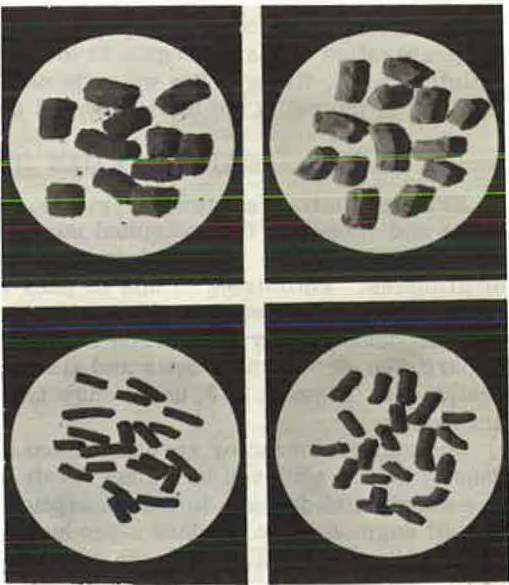


Figure 4. Unfired and fired fly ash aggregate made by extrusion process.

are used; either the material is made in different sizes and then blended, or, as in the case of extruded products, the distribution of the size may be established in the green bodies prior to the firing cycle. To produce finer aggregate, it is customary to crush the material, screen it, and proportion it so that it meets the requirements of the applicable specification for size gradation. For this reason, the fly ash aggregate plants usually incorporate crushing, screening, and blending operations in the manufacturing process.

#### PROPERTIES OF FLY ASH AGGREGATE

There is as yet no standard method to measure the strength of the individual granules, although several methods are used in individual laboratories. A study reported by the Central Electricity Generating Board (3) provides some results of tests of pellet strength made on aggregate utilizing different burning techniques. This report shows that the strengths are quite good for those pellets produced by the sintering grate, as well as those produced experimentally in rotary or shaft kiln equipment. In general, the aggregate produced from fly ash is evaluated on the basis of ASTM Specification C 330 and the reference test methods as called for in this specification. The tests include measurements of concrete-making properties, that is, strength and unit weights, drying shrinkage, popouts, and durability. Also of importance is the test for staining.

The firing process results in some sintering of individual particles of fly ash on the outer surface of the aggregate coupled with a more general fusion action in the interior. The sintered exterior normally represents less than 10 percent of the total volume. Figure 5 shows a close-up view of the typical structure of a cross section of fly ash aggregate. The structure of the exterior sintered portion of the aggregate results from the individual particles of the fly ash adhering to adjacent particles by partial melting or softening. The structure of the interior of the aggregate represents a

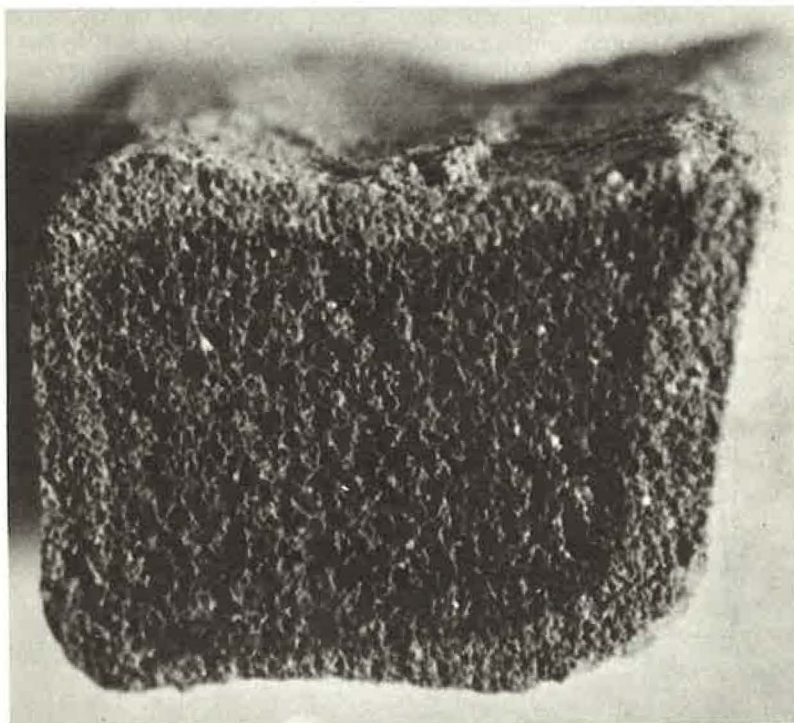


Figure 5. Close-up of a sawed particle of fly ash aggregate enlarged 10 times.

TABLE 2  
FLY ASH LIGHTWEIGHT AGGREGATE PROPERTIES

Test	Normal Range for Fly Ash Aggregate	ASTM Specification C 330
Unit weight, lb/ft <sup>3</sup> (kg/m <sup>3</sup> )		
At F. M. = 6.5 (nominal 3/4 in. to No. 4)	37 to 48 (593 to 769)	55 maximum
At F. M. = 5.8 (nominal 3/8 in. to No. 8)	41 to 51 (657 to 817)	55 maximum
At F. M. = 3.5 (nominal 3/8 in. to 0)	53 to 61 (849 to 977)	65 maximum
At F. M. = 3.0 (nominal No. 4 to 0)	56 to 63 (897 to 1,009)	70 maximum
24-hour absorption, percent	14 to 24	—
Staining	None to very light	—
Loss on ignition, percent	0 to 3.4	5.0 maximum

NOTE: F.M. means fineness modulus.

multicellular structure of a more completely melted product. In both cases the voids in the aggregate are caused by evaporation of the mixing water and elimination of carbon during the sintering process.

Table 2 gives the normal ranges of properties on a number of fly ash aggregates. It is to be noted that the values for absorption are quite high. The rate of absorption is also high and the effect that this has on compositions such as concrete is believed to be more beneficial than deleterious. It has been reported (4) that the water absorbed by this aggregate when preparing a batch of concrete is continuously available during the curing process. The aggregate can thereby provide curing water rather than absorbing water out of the cement matrix of the concrete mix. This may explain in part why these materials often provide somewhat superior performance when used in concrete. Field experience has indicated that plastic shrinkage of the concrete is also substantially reduced (presumably as a result of the highly saturated condition of the aggregate). The relative absorption of the different portions of the fly ash aggregate, as well as the uncrushed and crushed aggregate materials, is shown in Figure 6. This graph is representative of materials produced commercially at the Corson Plant at Plymouth Meeting, Pennsylvania.

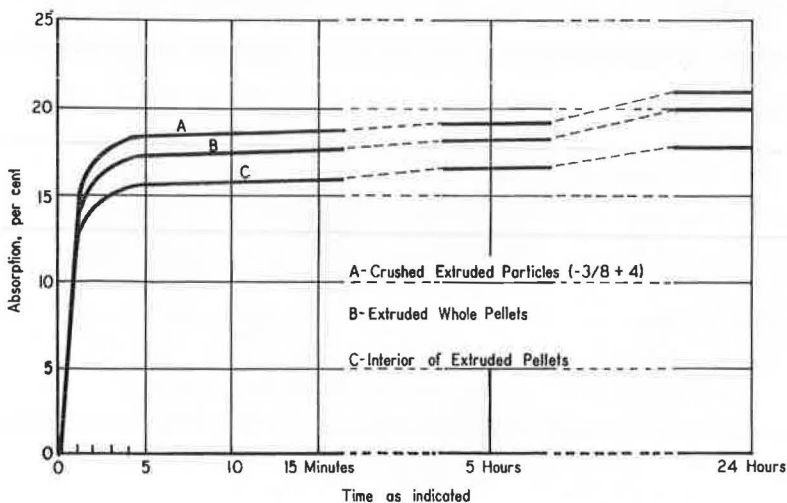


Figure 6. Moisture absorption of different fly ash aggregate products made from a single source of fly ash.



TABLE 3  
POZZOLANIC STRENGTH OF FINELY  
GROUND FLY ASH AGGREGATE<sup>a</sup>

Material Under Test	Blaine Fineness, $\text{cm}^2/\text{gm}$	Pozzolan Index, Percent of Control
Fly ash from source A	2,450	121
Ground lightweight aggregate made from source A fly ash	2,250	197
Fly ash from source B	5,420	130
Ground lightweight aggregate made from source B fly ash	5,330	151

<sup>a</sup>Modified test according to ASTM Method C 311-63 T.

With respect to the bulk density of aggregate produced from fly ash, some of the variation depends on the raw material from which the aggregate was produced. For instance, fly ash with very high fineness and low carbon content will usually result in a product having the higher densities. The data given in Table 2 illustrate the effect of aggregate size on bulk density.

Any residual fine fly ash particles that are either carried through the burning process or are formed by abrasive action of the aggregate during handling are still highly pozzolanic and will react in much the same manner when mixed with portland

cement as the unfired fly ash. This results in the usual long-term improvements in strength and dimensional stability of the concrete. When the lightweight aggregate produced from fly ash is pulverized to pass a No. 200 mesh sieve, virtually all of the original pozzolanic activity is recovered. Table 3 gives data on this effect as measured with two lightweight aggregate materials from different sources of fly ash. Figure 7 is a close-up of a fractured specimen of concrete containing fly ash aggregate. The excellent bond established at the interface between the aggregate and the mortar matrix is typical of results with fly ash material.

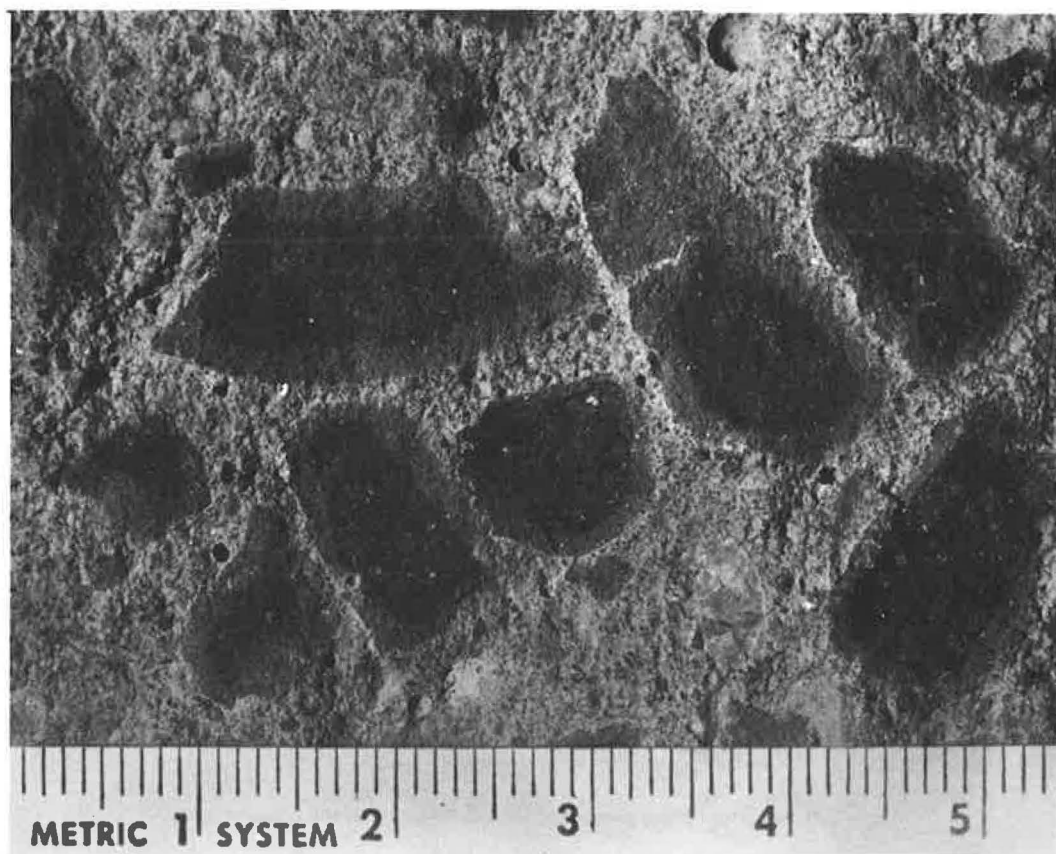


Figure 7. Fractured specimen of lightweight concrete containing fly ash aggregate.

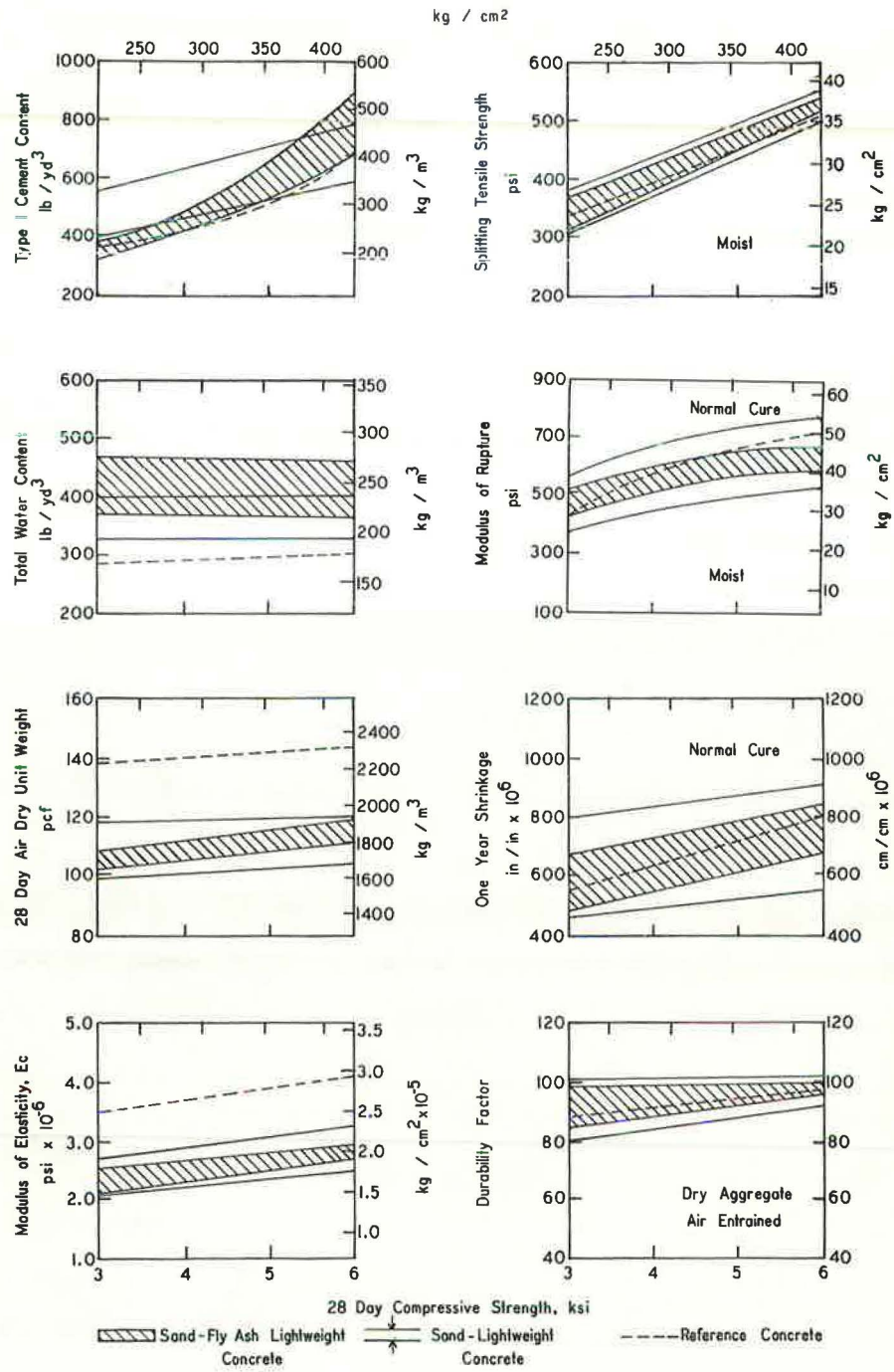


Figure 8. Comparison of lightweight concretes using sand as the fine aggregate.

Another important difference between aggregates produced from fly ash is in the shape of the individual granule. Uncrushed materials, such as those produced by the Corson process (which makes an extruded shape), have been demonstrated to have some advantage over the round pellets in improved workability and pumpability of the concrete. Crushed materials have been blended with sand and have been found to be effective in packaged concrete. The overall balance of properties, i.e., density, strength, and workability, in these latter mixtures is quite good.

## PROPERTIES OF HARDENED CONCRETE

Considerable structural concrete has been produced with lightweight aggregate made from fly ash. In general, the concrete has demonstrated satisfactory performance and compares very favorably with other forms of lightweight aggregate concrete. A recent report of Committee 213 of the American Concrete Institute (5) provides a good description of lightweight aggregate concrete and includes fly ash aggregate in the total evaluation made.

Figure 8 shows graphs describing concrete properties using fly ash aggregates produced in various plants throughout the world. The unshaded bands shown in the graphs are taken directly from the ACI report, as are the dotted lines representing a typical normal weight concrete. The shaded bands, as superimposed on this background, represent the fly ash lightweight aggregate concrete.

The graphs in Figure 8 are based on the use of normal weight natural sand as the fine aggregate. Most of the companies producing concrete commercially use this type of composition because of difficulties in workability experienced when lightweight aggregate is used in both the coarse and fine fractions.

Figure 9 shows data on the latter type mixtures. Because the source of this information is limited to fly ash aggregate concrete produced at the Corson Company plant, the pertinent data are given in the form of a single line rather than in a band, as is the case in Figure 8. For comparative purposes, Figure 9 also includes the background information as presented by ACI; the shaded band represents all lightweight aggregates and the dotted line again represents normal concrete.

A study recently reported by the Portland Cement Association Laboratories (6) provides additional information on fly ash aggregate concrete. The Portland Cement Association program has utilized material that has been supplied from a number of processing plants in this country. An interesting report of studies carried out in Yugoslavia (7) gives information on the feasibility of using fly ash from lignite coal to produce acceptable lightweight materials. A cooperative investigation is currently under way (8) to determine the fire-resistance of concretes produced with fly ash lightweight aggregate. An early report by Pearson and Asce (9) states that fire tests on fly ash aggregate concrete conducted for New York City, using ASTM Method E 119-62, shows that a nominal-size specimen of 4 by 12 by 60 in. has a fire-resistance of 3 hours.

Figure 10 provides some data on the thermal values for fly ash lightweight aggregate concrete. Included in this figure are data supplied through the courtesy of the Centre Scientifique et Technique du Bâtiment (10). The spread in the K-values as plotted reflects the conditions present at the time the measurements are made. The higher value of K at each density results primarily from tests of concrete made in a moist condition. There is considerable interest, in France as well as in other parts of the world, in the use of this type of concrete in building prefabricated structures. The very favorable K-values that are obtained, together with the high strength and other properties of the fly ash aggregate concrete, enable the fabricator to produce exterior walls and other units of minimum thickness and weight but of adequate strength.

Table 4 gives design criteria that are currently in use for commercial mixtures in this country. Comparison of these designs with the recommended practice, as developed by ACI 613A-59, indicate that the fly ash aggregate falls well within the ranges of that study. These design criteria provide adequate factors of safety when compared with the strength results as shown in Figures 8 and 9.

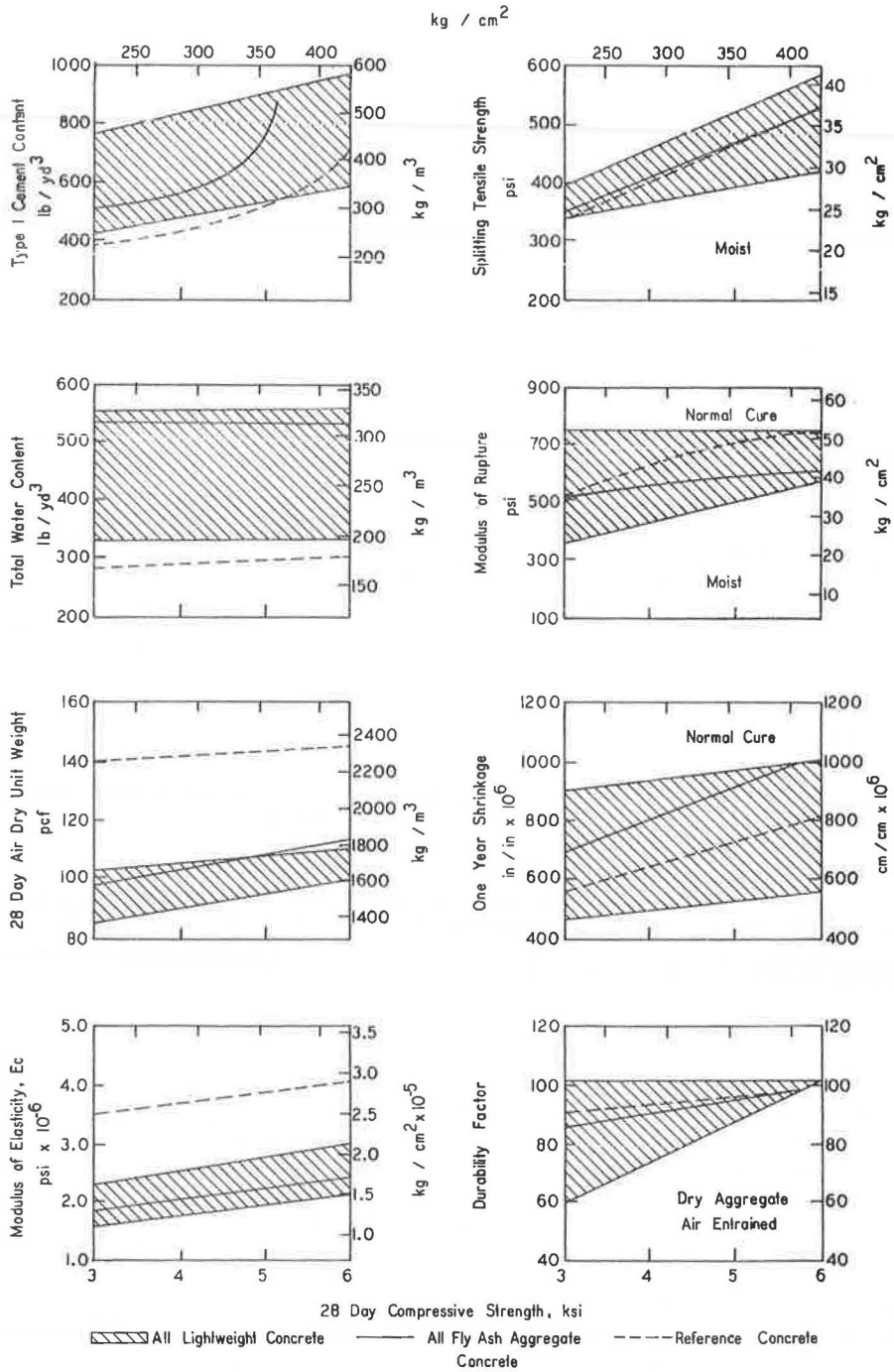


Figure 9. Comparison of concretes using all lightweight aggregate.

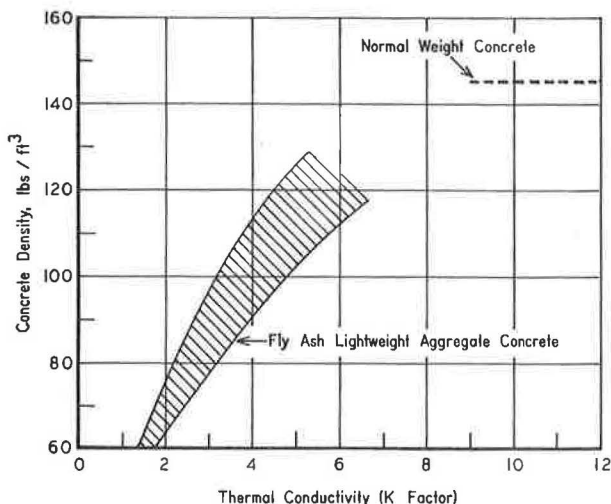


Figure 10. Range of thermal conductivities for fly ash aggregate concrete.

TABLE 4  
CONCRETE MIX DATA

Design Mix	Quantities Per Cubic Yard						Slump (in.)	Air-Entraining Agent (oz./sack)	Percent Air	Weight of Plastic Concrete (lb/cu ft)	Weight of Concrete, 28 Days (lb/cu ft)
	Cement		Aggregate (lb)		Water <sup>a</sup>						
			Corson-Lite, 3/4-in. Size, Bone Dry <sup>b</sup>	Concrete Sand, S. S. D. <sup>c</sup>							
	Sacks	Pounds			Pounds	Gallons					
1000	3.35	315	950	1,320	441	53	4	1.50	6	112	106
2000	5.00	470	875	1,300	433	52	4	1.75	6	114	109
3000	6.00	564	875	1,240	437	52.5	4	1.75	6	115	110
4000	7.00	658	875	1,180	441	53	4	1.75	6	116	111
5000	8.00	752	875	1,120	441	53	4	2.00	6	117	112

<sup>a</sup>Total water, including water of absorption.

<sup>b</sup>Based on Corson-Lite, 44 lb/cu ft density.

<sup>c</sup>S.S.D. = saturated surface dry.

## SUMMARY

Fly ash aggregate is being successfully used in structural concrete. Because of the light weight of the aggregate, it finds application for structural concrete in the range of 95 to 116 pcf (1,520 to 1,860 kg/cm<sup>3</sup>) and may also be used for insulating concrete at lower densities. With proper quality fly ash, commercial sintering processes produce various shapes of aggregate similar in properties to other structural lightweight aggregates. However, the high rate of absorption and residual pozzolanic properties provide some advantage over other materials. Tests of concrete using fly ash aggregate give results falling well within the range of values typical of lightweight concrete in general use.

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# Fired-Clay Aggregates for Use in Flexible Bases

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This is the second report issued under a research study dealing specifically with nonbloated synthetic aggregates produced from naturally occurring clays. The first report described the results of a laboratory investigation that indicated that most (if not all) highly plastic soils could be used to produce high-quality synthetic aggregates. The present report describes a later investigation directed toward the development of acceptance criteria that can be used to evaluate aggregates of this type. The investigation included the production of aggregates in the rotary kiln, the evaluation of these aggregates, and a study of several synthetic aggregate flexible bases that have been in service in the vicinity of Houston, Texas, for several years.

•THIS PROGRESS REPORT is the second part of a study entitled "Synthetic Aggregate Research" being conducted by the Texas Transportation Institute, sponsored by the Texas Highway Department and the U.S. Bureau of Public Roads. This phase deals specifically with synthetic aggregates for use in flexible bases, and its primary objective is to develop acceptance criteria for such aggregates. It was initiated in 1966 after nonbloated, fired-clay aggregates had been used experimentally in flexible bases on several Texas highways. To the author's knowledge, fired-clay aggregates are, at present, the only synthetic aggregates economically feasible for use in flexible bases. Therefore, all research efforts so far have been directed toward evaluating this type of aggregate.

The first study done under this phase of the research was an investigation into the chemical and physical stability of laboratory-produced aggregates. The results were reported in a paper by Moore et al. (1), which included the following tentative conclusions:

1. The clay minerals, montmorillonite, illite, and kaolinite, will not rehydrate under atmospheric conditions once they have been completely dehydrated (dehydroxylated); therefore, once they have been completely dehydrated, they become chemically stabilized for use as highway construction materials. Complete dehydration is accomplished by heating the clay and holding it at the elevated temperature for sufficient time to allow the dehydration to occur. A period of 15 minutes at 1400°F. was sufficient to completely dehydrate the clay present in the small, oven dry laboratory specimens made from the Texas soils investigated.

2. Incomplete dehydration of aggregates made by dehydrating clay-type soils can be detected by a relatively simple laboratory test.

3. Most (if not all) clay-type soils having a relatively high strength when air dried can be fired to produce hard, durable aggregates suitable for use in flexible base and asphaltic concrete.

The conclusions listed above were based almost entirely on an evaluation of cylindrical particles ( $\frac{1}{2}$  in. in diameter by  $1\frac{1}{2}$  in. long) that had been fired in a laboratory muffle furnace (Figs. 1 and 2). Therefore, the next investigation initiated under this phase of the research was an evaluation of aggregates produced in a rotary kiln, and the development of acceptance criteria for such aggregates. The investigation included the production of aggregates in the Texas Transportation Institute rotary kiln, their

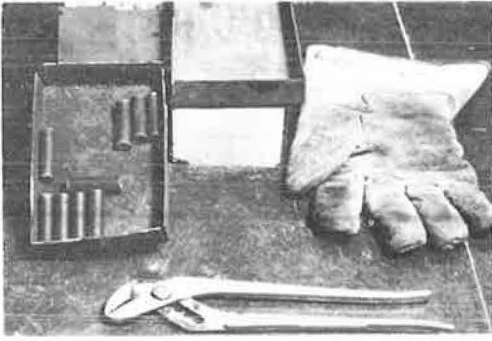


Figure 1. Molded specimens ready for firing in a laboratory muffle furnace.

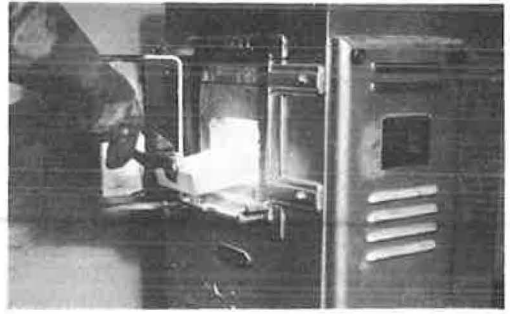


Figure 2. Specimens being removed from a laboratory muffle furnace after firing.

evaluation, and a study of several synthetic aggregate flexible bases that had been in service in the vicinity of Houston, Texas, for several years.

All research results to date are favorable to the use of fired-clay aggregates in flexible bases. The results indicate that these aggregates have great potential for use in highway construction in the many areas of the world where high-quality aggregates no longer exist, but where the clays required for the production of synthetic aggregates are plentiful.

#### MATERIALS

As a step in the development of a preliminary recommended criterion for the acceptance of flexible base aggregates, the Texas Transportation Institute's research rotary kiln (Fig. 3) was used to produce several samples of graded aggregates. The development of the research kiln and its capabilities are described elsewhere (2, 3).

Aggregate samples were produced from three plastic soils obtained from different sources in Brazos County, Texas. The Atterberg limits and gradations of the soils used are given in Table 1. Standard Texas Highway Department test procedures were followed in these determinations. Aggregates were made by firing each of the three soils at 8 different temperatures; thus, 24 aggregate samples were available for testing. Each sample consisted of about 250 lb of graded material, approximately 95 percent of which was sized between the 1-in. sieve and the No. 10 sieve.

In addition to aggregates made by the Texas Transportation Institute, 4 aggregate samples that had been produced in commercial rotary kilns for use in flexible bases were obtained. Two of the 4 aggregates have been successfully used in flexible bases by the Texas Highway Department. These are referred to as the Hopkins aggregate used by the Paris district and the Wharton aggregate used by the Houston district. Two other samples, designated as the Madison 1 and Madison 2 aggregates, were obtained. These aggregates had been investigated by the Bryan district for possible future use in flexible bases. The results of laboratory tests conducted by the Bryan district, which were

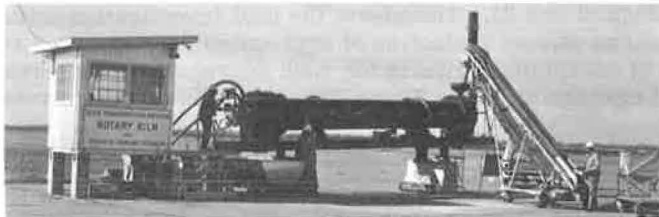


Figure 3. Texas Transportation Institute's research rotary kiln.

TABLE 1  
MATERIALS USED FOR RESEARCH AGGREGATE PRODUCTION

Raw Material	Liquid Limit	Plastic Limit	Plasticity Index	Gradation <sup>a</sup>		
				Percent Sand	Percent Silt	Percent Clay
Red clay	74	28	46	4	24	72
Gray clay	67	23	44	5	41	54
Black clay	53	19	34	13	34	53

<sup>a</sup>Determined by hydrometer analysis (MIT classification: sand, 2 to 0.06 mm; silt, 60 to 2 microns; and clay, smaller than 2 microns).

reported by Long (4), indicated that either of the two aggregates would be suitable for use in flexible bases.

None of the above aggregates can be classified as lightweight synthetic aggregates. However, 5 samples of lightweight aggregate—i.e., having a unit weight of less than 55 pounds per cubic foot (pcf)—produced commercially in Texas primarily for use in structural concrete were obtained. These aggregates are currently under investigation for portland cement concrete application in the first part of this study.

In summary, 33 aggregate samples were investigated; 24 of them were produced in the TTI research kiln, 2 have been used successfully in flexible bases on Texas highways, 2 have been investigated for possible similar future use, and the remaining 5 are lightweight aggregates that are available commercially in Texas.

EVALUATION OF AGGREGATES

The simple testing procedure developed during the initial laboratory studies is shown in Figures 4 and 5. Basically it consists of the cooking of specimens under water in a common kitchen-type pressure cooker, and then observing the effect of such treatment on the particles. This procedure works quite well for laboratory-produced cylindrical specimens because deterioration caused by the treatment is readily apparent on the smooth surface of the particles. It was initially thought that the same procedure would yield significant changes in the gradation of kiln-produced aggregates if they were not sufficiently dehydroxylated; however, this was found not to be the case. Only minor changes in gradation were observed after the cooking treatment for many aggregates obviously unsuitable for use in flexible bases. After the cooking treatment, these aggregate samples could be easily crumbled with the fingers.

Several variations of the initial test were tried. A procedure found to be adequate is given in Appendix A. It is called the pressure-slaking test to distinguish it from the slaking test (now considered obsolete) described elsewhere (1) and from the slaking procedures often used in the preparation of soil samples for laboratory testing. Basically the pressure-slaking test consists of the same underwater pressure-cooking (Fig. 6), but an additional treatment of severe agitation in water is inserted between the cooking and the gradation-change measurement. The agitation is accomplished using



Figure 4. Specimens prepared for cooking in a pressure cooker.

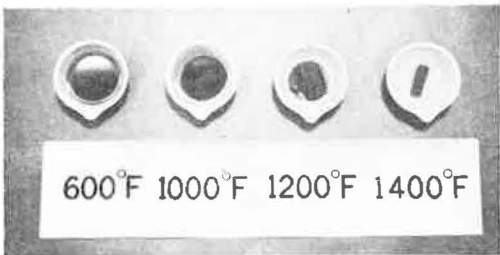


Figure 5. Specimens after cooking in pressure cooker.



Figure 6. Bottles containing kiln-produced aggregates after cooking in pressure cooker.



Figure 8. Complete samples immediately after cooking and subsequent agitation in water (numbers refer to approximate firing temperature, hundreds of degrees F).

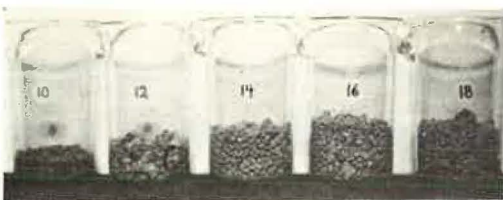


Figure 9. Fractions of samples retained on the No. 40 sieve.

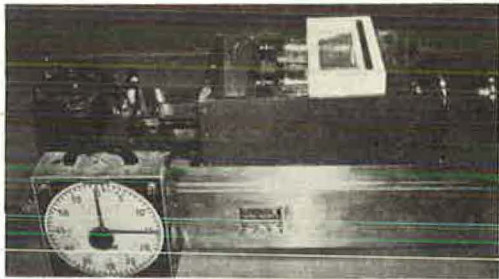


Figure 7. Kiln-produced aggregates subjected to severe agitation in water by a heavy-duty shaker.

TABLE 2  
TEST RESULTS FOR RESEARCH AGGREGATES AND AGGREGATES PRODUCED IN COMMERCIAL KILNS

Aggregate Type	Sample Designation <sup>a</sup>	Pressure Slaking <sup>b</sup> (percent loss)	Los Angeles Abrasion <sup>c</sup> (percent wear)
Red clay, nonbloated, produced by TTI	RC-1000	66.6	64.2
	RC-1100	38.9	61.3
	RC-1205	20.5	56.6
	RC-1295	6.2	50.2
	RC-1390	4.5	42.8
	RC-1585	2.2	36.1
	RC-1800	2.5	36.8
	RC-1910	2.8	33.9
Gray clay, nonbloated, produced by TTI	GC-1030	66.4	62.2
	GC-1095	61.7	66.6
	GC-1200	25.6	55.2
	GC-1305	13.8	61.5
	GC-1400	9.8	62.8
	GC-1600	6.1	49.4
	GC-1800	5.8	52.7
	GC-1930	4.0	39.6
Black clay, nonbloated, produced by TTI	BC-1010	55.9	71.3
	BC-1100	37.4	67.0
	BC-1205	16.8	64.9
	BC-1310	9.3	60.1
	BC-1395	6.7	56.8
	BC-1610	6.5	51.7
	BC-1800	4.2	37.4
	BC-1940	4.2	37.0
Nonbloated, produced commercially	Hopkins	8.3	35.0
	Wharton	9.7	43.6
	Madison 1	6.9	33.3
	Madison 2	7.7	38.3
Bloated, lightweight, produced commercially	R	2.7	27.5
	S	4.9	22.5
	C	5.7	40.4
	E	2.5	25.2
	D	5.0	23.1

<sup>a</sup>The 4-digit numbers shown in some sample designations refer to the maximum kiln temperature measured during sample production.  
<sup>b</sup>Each pressure-slaking loss value is the average of three tests performed in accordance with Appendix A.  
<sup>c</sup>Each Los Angeles abrasion value is the result of a single test (ASTM Method C 131).



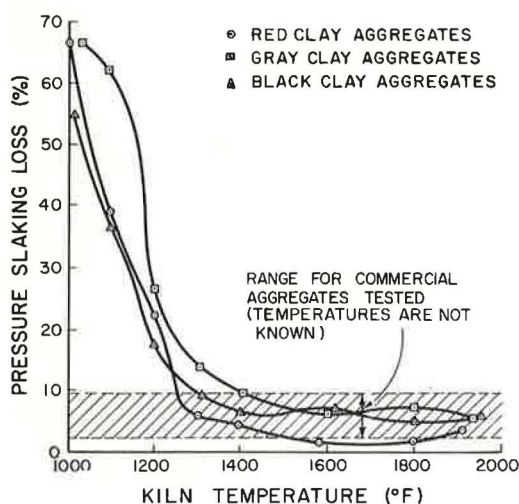


Figure 10. Pressure-slaking loss versus maximum kiln temperature for research aggregates made from three clays.

consisted of a mixture of approximately 70 percent aggregate produced from clay in a rotary kiln and 30 percent field sand. The Wharton aggregate (given in Table 2 for comparison with the research aggregates) was used in at least one of these projects. The field sand, taken from several sources, was required to have a liquid limit of less than 35 and a plasticity index of less than 10. In one of the projects, the base material was stabilized with lime. According to local engineers, all of the projects are still in good shape. One short section has been reworked (necessitated by a bridge grade change) and the synthetic aggregate flexible base in this section was salvaged and reused.

In the summer of 1968, at 6 different locations several miles apart, sections of these synthetic aggregate flexible bases were sampled and field-tested (Fig. 11). Samples from 5 of these locations were prepared for laboratory testing in accordance with standard Texas Highway Department testing procedures.

As may be seen from the gradations given in Table 3, the amount of soil binder (fraction finer than No. 40 sieve) in the 5 samples prepared for laboratory testing varied from 22 to 34 percent and averaged 28 percent. When one considers that the kiln-produced aggregates had from 2 to 5 percent soil binder, and that the construction specifications required that they be mixed with 30 percent of a field sand with 99 percent soil binder, it appears that there has been no significant disintegration of synthetic aggregates during the 5 to 6 years they have been in service.

The pressure-slaking tests (Table 3) were made on aggregate samples separated from the base samples by washing over the No. 10 sieve. The loss values obtained varied from 8.3 to 15.5 percent and averaged 11.0 percent, as compared to a range of 6.9 to 9.7 percent and an average of 8.2 percent for the 4 commercial flexible base aggregates sampled from stockpiles

a standard laboratory heavy-duty shaker (Fig. 7). Typical results of cooking and gradation are shown in Figures 8 and 9. One can observe in these figures the relative amounts of disintegration resulting from the test.

Results of the pressure-slaking test and the Los Angeles abrasion test for the 24 aggregates produced by TTI as well as the 9 aggregates produced commercially are given in Table 2 and shown in Figure 10. From the figure it is clear that the research aggregates made at about 1,000 F would not be suitable for use in a flexible base, whereas all those made at firing temperatures of 1,400 F and higher compare favorably with the synthetic aggregates produced commercially.

### EVALUATION OF FIELD SAMPLES

During 1963 and 1964, the Houston district of the Texas Highway Department constructed several projects totaling about 15 miles in length utilizing synthetic aggregate flexible base. The base material



Figure 11. Synthetic flexible bases sampled for laboratory testing after several years of service in the Houston area.



TABLE 3  
TEST RESULTS OBTAINED ON SAMPLES FROM HIGHWAYS NEAR HOUSTON

Section No.	Moisture (percent)	Dry Density (pcf)	Percent Retained by Sieves				Pressure-Slaking Test
			3/4 in.	No. 4	No. 10	No. 40	
2	13.5	111.2	0	29	50	66	12.6
7	15.8	105.9	1	32	60	78	10.0
8	15.9	90.6	0	28	54	71	8.7
9	15.8	90.8	2	34	58	73	15.5
14	15.7	111.4	1	24	49	72	8.3
15 <sup>a</sup>	16.4	107.0	—	—	—	—	—

<sup>a</sup>Base material was stabilized with lime and could not be tested.

(Table 2). Thus, the material samples from the roadway had a somewhat greater range and a higher average loss than was the case for the materials sampled from stockpiles. According to the general trend shown in Figure 10, it appears that the synthetic aggregates taken from the roadway probably were produced at temperatures slightly lower than those taken from stockpiles.

In summary it can be said that all of the synthetic aggregate bases appear to have performed satisfactorily to date. The laboratory tests made on the samples that had been in service for several years indicate that these aggregates had not undergone any significant disintegration. Thus, it can be concluded—at least for the Houston environment—that synthetic aggregates suitable for use in flexible base can be produced by firing highly plastic clays. Such aggregates may or may not be suitable for use in more severe environments.

In the opinion of the author, 10 percent loss determined by the pressure-slaking test (Appendix A) is a safe and reasonable upper limit for an acceptance criterion for flexible base synthetic aggregates in Texas to ensure that the aggregates have been fired sufficiently to remain stable in their intended use.

PRESSURE-SLAKING TEST, MOD. 1

In order to utilize existing equipment in Texas Highway Department district laboratories, a modification of the pressure-slaking procedure described in Appendix A was undertaken. The modified procedure is called the "Pressure-Slaking Test (Mod. 1)" and is described in Appendix B. Basically it consists of the same procedure used in the pressure-slaking test except that it uses a Tyler sieve shaker (Figs. 12 and 13) for accomplishing the agitation in water instead of the heavy-duty shaker (Fig. 7). Comparative

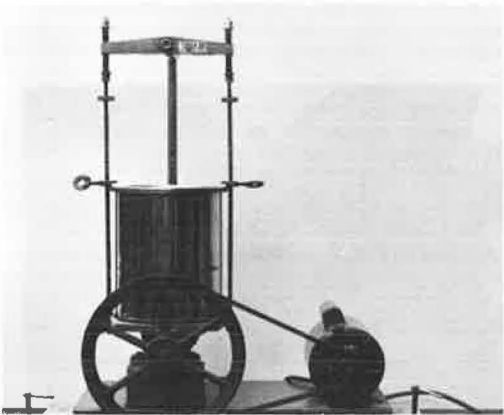


Figure 12. A Tyler sieve shaker used in the modified pressure-slaking test instead of heavy-duty shaker shown in Figure 7.



Figure 13. Container designed to fit shaker for agitating 5 samples simultaneously.

TABLE 4  
COMPARATIVE RESULTS OF PRESSURE-SLAKING TEST  
VERSUS PRESSURE-SLAKING TEST (MOD. 1)

Aggregate Designation	Pressure-Slaking Test, Percent Loss <sup>a</sup>				Pressure-Slaking Test (Mod. 1), Percent Loss <sup>b</sup>			
	Test 1	Test 2	Test 3	Average	Test 1	Test 2	Test 3	Average
RC-1000	60.3	60.4	79.0	66.6	53.6	47.3	43.3	48.1
RC-1100	37.4	33.2	46.0	38.9	21.9	26.7	23.1	23.7
RC-1205	22.9	18.7	20.0	20.5	9.4	7.6	9.8	8.9
RC-1295	5.9	5.6	7.0	6.2	2.1	2.2	2.5	2.3
RC-1390	4.0	4.4	5.0	4.5	1.5	1.6	1.3	1.5
RC-1585	1.8	1.9	3.0	2.2	1.3	1.0	0.8	1.0
RC-1800	2.4	2.1	3.0	2.5	1.2	0.8	1.0	1.0
RC-1910	2.3	2.1	4.0	2.8	0.9	1.2	0.6	0.9
GC-1030	66.6	65.5	67.2	66.4	54.3	60.3	52.6	55.7
GC-1095	62.7	60.7	—	61.7	51.6	46.0	44.6	47.4
GC-1200	27.5	26.2	23.1	25.6	13.0	12.8	14.1	13.3
GC-1305	12.7	14.9	—	13.8	5.8	4.8	5.6	5.4
GC-1400	10.2	9.2	10.0	9.8	3.9	3.7	4.5	4.0
GC-1600	6.9	5.5	6.0	6.1	1.9	1.9	2.5	2.1
GC-1800	6.4	5.1	6.0	5.8	2.0	2.1	2.1	2.1
GC-1930	4.0	3.1	5.0	4.0	1.9	1.4	1.0	1.4
BC-1010	51.6	52.2	64.0	55.9	35.2	36.1	32.4	34.6
BC-1100	36.0	36.3	40.0	37.4	23.9	22.7	19.5	22.0
BC-1205	17.4	16.1	17.0	16.8	7.0	7.4	9.0	7.8
BC-1310	8.9	10.0	9.0	9.3	4.1	3.6	3.2	3.6
BC-1395	6.5	7.0	6.5	6.7	2.2	2.2	2.8	2.4
BC-1610	6.1	6.4	7.0	6.5	2.5	2.7	2.6	2.6
BC-1800	3.3	4.3	5.0	4.2	1.4	1.6	1.8	1.6
BC-1940	4.1	4.6	4.0	4.2	0.9	1.1	1.4	1.1

<sup>a</sup>Analysis of variance: within sample standard deviation, 3.1; within sample CV, 16.0 percent.

<sup>b</sup>Analysis of variance: within sample standard deviation, 1.8; within sample CV, 14.5 percent.

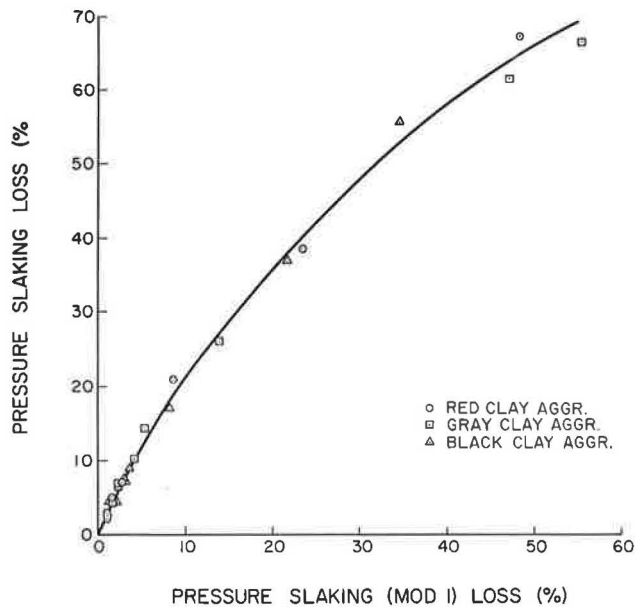


Figure 14. Comparison between original and modified pressure-slaking test results.

test results obtained using the two different procedures on the 24 aggregate samples produced by TTI are given in Table 4. It can be seen from these data that results obtained using the modified test are always lower than those obtained using the original procedure.

Comparative analyses of variance for the data obtained from the two test procedures indicate that the repeatability of the two is about the same. A plot of comparative values is shown in Figure 14. From this it is clear that there is a strong correlation between the tests; thus, the two are essentially measuring the same property. Specifically, it can be said that the recommended acceptance criterion of 10 percent loss based on the pressure-slaking test is equivalent to a loss of 4 percent based on the modified test.

### CONCLUSION AND RECOMMENDATIONS

The following findings were reached as a result of the investigation described in this report:

1. Synthetic aggregates suitable for use in flexible bases in Texas can be produced in rotary kilns from highly plastic clays. These aggregates may or may not be suitable for use in locations having a more severe environment than Texas, such as locations subjected to deep frost penetration.
2. Incomplete dehydration of synthetic aggregates made by firing clays can be detected by two relatively simple tests. The procedures for these tests are given in Appendixes A and B.
3. Test results and service records to date indicate that these aggregates have great potential for use in highway construction in the many areas of the world where high-quality aggregates no longer exist but where the clays required for the production of synthetic aggregates are plentiful.

Based on the results of this investigation, it is recommended that synthetic aggregates for use in Texas flexible bases have a loss measured by the pressure-slaking test (Appendix A) of less than 10 percent or a loss measured by the modified pressure-slaking test (Appendix B) of less than 4 percent.

### ACKNOWLEDGMENTS

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The opinions, findings, and conclusions expressed in this paper are those of the author and not necessarily those of the Bureau of Public Roads.

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## *Appendix A*

### PRESSURE-SLAKING TEST

#### Scope

The test method described here is intended to be used to evaluate the amount of dehydration that has occurred in the production of synthetic aggregates fired in a rotary kiln. This procedure is a modification of the procedure previously reported elsewhere (1).

#### Apparatus

The apparatus shall consist of the following: (a) pressure cooker (common kitchen-type with 6-quart capacity and 15-psi pressure regulator); (b) centrifuge bottles, 500 ml Pyrex; (c) balance with 3,000 gram capacity having a sensitivity of 0.1 gram; (d) heavy-duty reciprocating laboratory shaker (Precision Scientific Cat. No. 5855 or equivalent); (e) sieves ( $\frac{3}{4}$ -in., No. 10, and No. 40 sieves that meet the requirements of ASTM Specification E 11); and (f) drying oven capable of heating to 105 C (220 F).

#### Sample

An unwashed representative sample of sufficient volume to half fill the centrifuge bottle should be chosen. The sample material is that which passes a  $\frac{3}{4}$ -in. sieve and is caught on a No. 10 sieve. Any material retained on the  $\frac{3}{4}$ -in. sieve should be crushed to pass this sieve using a minimum amount of crushing. Because synthetic aggregates vary widely as to specific gravity, a volumetric measure of the sample is used rather than weight.

#### Procedure

1. Place the sample into the centrifuge bottle, and add 200 ml of distilled water. It is not necessary to determine the initial weight of the sample. (Repeat for any number of samples up to as many as can be conveniently placed in the pressure cooker.)
2. Place the centrifuge bottles containing the aggregates in the pressure cooker, add approximately  $\frac{1}{2}$  in. of distilled water to the pressure cooker, and seal the lid tightly.
3. Heat the pressure cooker with a large Bunsen burner until full pressure is indicated by the pressure regulator.
4. Adjust flame to allow only a slight escape of steam and maintain pressure for 15 minutes. Remove the Bunsen burner, release the pressure, and remove the centrifuge bottles.
5. After cooling to approximately 100 F, place corks in the centrifuge bottles and place the bottles in the laboratory shaker. Shake the aggregates for 15 minutes.
6. After removing the bottles from the shaker, wash the sample over a No. 40 sieve, taking care not to lose any of either -40 or +40 material.
7. Dry both -40 and +40 material to a constant weight at 105 C (220 F). Because of rehydration, the final total weight of the sample may be greater than the initial weight.



### Calculations

The pressure-slaking loss is expressed as the percent passing the No. 40 sieve and is calculated by the following equation:

$$\text{Percent Loss} = \frac{\text{Weight of minus-40 mesh material}}{\text{Total weight of material}} \times 100$$

## ***Appendix B***

### **PRESSURE-SLAKING TEST (MOD. 1)**

#### Scope

This procedure is a modification of the pressure-slaking test procedure given in Appendix A. The modification was made to better utilize existing equipment in the Texas Highway Department district laboratories.

#### Apparatus

The apparatus shall consist of the following: (a) pressure cooker (common kitchen-type with 6-quart capacity and 15-psi pressure regulator); (b) centrifuge bottles, 500 ml Pyrex; (c) Tyler sieve shaker (Soiltest Model No. C1-305A or equivalent, cpm =  $285 \pm 10$ , throw =  $1\frac{3}{4} \pm \frac{1}{4}$  in.); (d) balance with 3,000 gram capacity having a sensitivity of 0.1 gram; (e) stainless steel bucket (fits Tyler sieve shaker) consisting of Bain Marie pot with cover and beaker without a pouring lip ( $8\frac{1}{4}$  quart, 8-in. body diameter,  $9\frac{3}{4}$ -in. depth,  $8\frac{3}{4}$ -in. over bead diameter, available from Texas Highway Department D-4 stock; (f) spacer ( $7\frac{3}{4}$ -in. diameter by 2 in. thick), rubber cushion ( $7\frac{3}{4}$ -in. diameter by  $\frac{1}{8}$  in. thick), and miscellaneous rubber sheeting or rags; (g) sieves ( $\frac{3}{4}$ -in., No. 10, and No. 40 sieves that meet the requirements of ASTM Specification E 11); and (h) drying oven capable of heating to 105 C (220 F).

#### Sample

An unwashed representative sample of sufficient volume to half fill the centrifuge bottle should be chosen. The sample material is that which passes a  $\frac{3}{4}$ -in. sieve and is caught on a No. 10 sieve. Any material retained on the  $\frac{3}{4}$ -in. sieve should be crushed to pass this sieve using a minimum amount of crushing. Because synthetic aggregates vary widely as to specific gravity, a volumetric measure of the sample is used rather than weight.

#### Procedure

1. Place the sample into the centrifuge bottle, and add 200 ml of distilled water. It is not necessary to determine the initial weight of the sample. (Repeat for any number of samples up to as many as can be conveniently placed in the pressure cooker.)

2. Place the centrifuge bottles containing the aggregates into the pressure cooker, add approximately  $\frac{1}{2}$  in. of distilled water to the pressure cooker, and seal the lid tightly.

3. Heat the pressure cooker with a large Bunsen burner until full pressure is indicated by the pressure regulator.

4. Adjust flame to allow only a slight escape of steam and maintain pressure for 15 minutes. Remove the Bunsen burner, release the pressure, and remove the centrifuge bottles.

5. After cooling to approximately 100 F, place corks in the centrifuge bottles and place the bottles vertically in the stainless steel bucket. (The rubber cushion should be



placed beneath the bottles and the rubber sheeting or rags inserted between the bottles to press them firmly against the side of the bucket.)

6. Place the spacer over the rubber corks in the bottles and fasten the cover to press the bottles against the bottom of the bucket.

7. Lock the stainless steel bucket in the Tyler sieve shaker and shake the aggregates for 15 minutes.

8. After removing the bottles from the shaker, wash the sample over a No. 40 sieve, taking care not to lose any of either -40 or +40 material.

9. Dry both -40 and +40 material to a constant weight at 105 C (220 F). Because of rehydration, the final total weight of the sample may be greater than the initial weight.

#### Calculations

The pressure-slaking (Mod. 1) loss is expressed as the percent passing the No. 40 sieve and is calculated by the following equation:

$$\text{Percent Loss} = \frac{\text{Weight of minus-40 mesh material}}{\text{Total weight of material}} \times 100$$

# An Evaluation of the Bulk Specific Gravity for Granular Materials

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Accurate determination of bulk specific gravity of aggregates is of paramount importance for successful design of bituminous paving mixtures as well as portland cement concrete. The complicating element in specific gravity determination is the achievement of saturated surface-dry conditions. The various modifications suggested by several investigators either offer little improvement or are too elaborate to be of practical value in the field or common laboratory. In this study, the bulk specific gravities of crushed aggregates and cylindrical rock cores from six limestone quarries, one slag, one crushed trap rock, and one synthetic aggregate (Synopal) were determined by five methods. The methods are standard ASTM, geometrical measurement, mercury displacement, and two chemical indicator methods proposed by the authors. The results by the standard ASTM methods, in terms of reasonableness and repeatability, are compared and evaluated in relation to results by the other four methods. The procedures of the proposed chemical indicator methods (cobalt chloride and fluorescein sodium salt) are described. The repeatability and reproducibility of the cobalt chloride indicator method are presented.

•**BULK SPECIFIC GRAVITY** is 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 ( $V_s$ ), the volume of the permeable voids ( $V_p$ ), and the volume of the impermeable voids ( $V_i$ ) times the unit weight of water ( $\gamma_w$ ):

$$\text{bulk specific gravity} = \frac{A}{(V_s + V_p + V_i) \gamma_w}$$

or

$$= \frac{A}{B - C}$$

where  $B$  is the saturated surface-dry weight (in grams) of the material in air and  $C$  is the weight (in grams) of saturated material in water. Those voids that cannot be filled with water after a 24-hour soaking are referred to as impermeable voids. Voids that can be filled with water after a 24-hour soaking period are referred to as permeable.

Percent voids is widely 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 necessary to calculate the voids properties accurately.

Bulk specific gravity is also used in design calculations of concrete mixtures. The specific gravity of aggregates must be known to enable the designer to compute the amounts of the various ingredients in the mixture. Because almost all batching is now done on a weight basis, the importance of correctness of its value is readily apparent.

Experience has shown that, for some materials, the results of bulk specific gravity are very difficult to reproduce. This is mainly caused by the personal element involved in judging the point at which the wet aggregate has dried out sufficiently to reach the "saturated surface-dry condition," which is theoretically the point at which all the surface moisture has gone from the particles but with the permeable pores remaining completely filled.

ASTM Standard Methods C 127 and C 128 outline means for determining absorption and bulk specific gravity of aggregates. These standards call for immersion of material in water for 24 hours, followed by drying until the surface-dry state is attained. Coarse aggregates are rolled in an absorbent cloth until all visible films of water are gone.

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 after removal of the cone, it is assumed to be in a saturated surface-dry state. In addition to variation caused by individual judgment, the fact that large particles tend to dry more quickly than small particles may lead to overdrying of the larger particles, unless the precaution is taken of recognizing and handling these particles separately. These variations become significant in the case of aggregates having a rough and porous surface.

For natural, well-graded fine aggregates, the saturated surface-dry condition is usually reproducible. 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 of practical value in the field or average laboratory.

Bulk specific gravity has also been determined by simple mensuration; for example, by use of a micrometer or a dial gage to determine the volume of regular shape samples. When the sample is irregular or particulate it has been determined by mercury displacement under a given pressure, provided the mercury does not wet the surfaces of the sample. Both methods have been included in this study to assess the comparative values of bulk specific gravity as obtained from ASTM standard methods. Rock cores from the same block samples have been used for both these methods to eliminate errors caused by sample variation.

A new chemical indicator method has been tried in this study. Its adoption is suggested for determining the saturated surface-dry condition of both coarse and fine aggregates. This seems very practical and eliminates most of the personal judgment involved in standard ASTM tests.

## OBJECTIVES

The purposes of this investigation are (a) to evaluate the reasonableness and repeatability of the standard ASTM methods for bulk specific gravity, as compared with other alternative methods; (b) to develop new, simple methods to determine the bulk specific gravity or the saturated surface-dry condition for granular materials more reliably; and (c) to evaluate these new methods as compared to the ASTM methods.

TABLE 1  
LIMESTONE AGGREGATES STUDIED

Number	County	Quarry	Beds or Ledges	Geological Formation
1	Adair	Menlo	3-6-Argentine	Missourian series, Pennsylvanian system
2	Blackhawk	Pints	Rapid	Cedar Valley formation, Devonian system
3	Hardin	Alden	—	Gilmore City formation, Mississippian age
4	Scott	Linwood	Davenport	Devonian system
5	Story	Cook	—	St. Louis formation, Mississippian series
6	Washington	Keota	Beds 14 through 22	Osage series, Mississippian age

## MATERIALS

### Crushed Aggregates

Six limestone aggregates that were obtained from various areas in Iowa and that range from most absorptive to least absorptive in character have been included in the study for both coarse and fine aggregates. Mercury-intrusion porosity (21-0.05  $\mu$  pore radius range) varied from 0.90 percent in Linwood limestone to 17.44 percent in Cook limestone, with varying pore characteristics and surface texture. Besides these, one trap-rock aggregate and one blast-furnace slag were included to provide variation in water absorption, pore characteristics, and specific gravity. Subsequently, at a later stage, Synopal (a synthetic aggregate manufactured in Denmark) was also included because it is supposed to have uniform structure and other characteristics and it could be used in the comparative study on bulk specific gravity. The details of limestone aggregates studied are given in Table 1. For coarse aggregates, the fraction passing the  $\frac{3}{8}$ -in. sieve and retained on the No. 4 sieve was used so as to have uniformity in results. For fine aggregates, the fraction passing the No. 4 sieve and retained on the No. 100 sieve was used (Table 2).

### Rock Cores

Rock cores  $\frac{1}{2}$  in. in diameter were drilled from blocks obtained from the respective limestone quarries to be used for determination of bulk specific gravity. They do not necessarily represent the beds from which the aggregates were procured but have been used to ensure material homogeneity and to assess the relative values of ASTM bulk specific gravity in comparison with that obtained from other methods. For uniformity, the rock cores were drilled at right angles to the natural bedding planes. For geometrical measurements, the ends of the rock cores were trimmed so as to have a true cylinder for accurate measurement of volume.

TABLE 2  
GRADATION OF FINE AGGREGATES

Sieve No.	Percent Passing					
	Menlo	Pints	Alden	Linwood	Cook	Keota
4	100	100	100	100	100	100
8	20	67	53	60	70	86
30	1	20	15	12	25	40
50	0.6	8.9	7.4	4.9	10.8	10.0
100	0.3	0.5	0.4	0.4	1.1	0.3

## Chemical Indicators

For the new methods to determine saturated surface-dry condition, four chemical indicators were tried: (a) cobalt chloride, (b) fluorescein disodium salt, (c) cupric chloride, and (d) copper sulfate. Of these, only the first two gave encouraging results and have been included in this study.

## METHODS FOR DETERMINING BULK SPECIFIC GRAVITY

### Rock Cores

ASTM Method—ASTM Standard Method C 127 was adopted for  $\frac{1}{2}$ -in. diameter rock cores to determine bulk specific gravity. The rock cores could be rolled uniformly on absorbent cloth to achieve a saturated surface-dry condition as defined by ASTM.

Geometrical Mensuration Method—In the geometrical mensuration method each rock core was weighed to 0.0001 gram and its dimensions were measured with a caliper up to 0.01 cm. Average diameter and length were calculated after measuring at three or four places. Duplicate determinations were made for each rock type. Bulk specific gravity was calculated as weight divided by measured volume.

Mercury Displacement Method—A mercury porosimeter, as used in determination of mercury-intrusion porosity and pore-size distribution of aggregates, was used to determine the bulk volume of the rock core at 5 psi. The theory and operation of the apparatus are described elsewhere (6, 7). The authors have devised a simpler apparatus, using a modified 250-ml burette connected to a sample chamber at one end and a mercury reservoir at the other end, to measure bulk specific gravity of aggregates by the mercury displacement method, but it will not be discussed here.

### Crushed Aggregates

ASTM Methods—ASTM Standard Methods C 127 and C 128 were followed for determination of bulk specific gravity of coarse and fine aggregates respectively, except that Chapman flasks were used for fine aggregates. The bulk specific gravity (Gb) was calculated as follows:

$$G_b = \frac{A}{V - 200}$$

where A = weight in grams of oven-dry sample in air, and

V = combined volume in millimeters of saturated surface-dry sample and water.

Mercury Displacement Method—The procedure used is the same as described for rock cores except that crushed aggregates were used instead in the sample chamber.

Chemical Indicator Methods—With the salt cobalt chloride there is a color change from the red hexahydrate through the violet monohydrate to the blue anhydrous salt; that is, anhydrous  $\text{CoCl}_2$  is blue whereas  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  is red. Use is made of this property of the indicator to pinpoint the saturated surface-dry condition. A 5 percent solution of cobalt chloride is prepared and the aggregate (coarse or fine) is immersed in this solution. After 24 hours of immersion, the aggregates are removed from the solution and spread on a table with a white top or in any wide white enameled tray. The aggregate may or may not acquire a pink tint, but as soon as drying proceeds and its surface is dry, it attains a distinct bluish color. This is assumed to be the saturated surface-dry condition. The remaining procedure is the same as indicated in ASTM Standard Methods C 127 and C 128. The detailed procedure is described in the Appendix.

In the other chemical indicator method, a solution (0.5 percent) of fluorescein sodium salt is prepared and the aggregate (coarse or fine) is immersed in this solution for 24 hours. When the aggregate is taken out of the solution, it acquires a distinct light yellow color. As the aggregate is spread on a white porcelain surface and is subjected to drying, the color of the aggregate changes from light yellow to distinct orange, thus achieving the saturated surface-dry condition. The remaining procedure is the same as in ASTM Standard Methods C 127 and C 128. The detailed procedure is described in the Appendix.



TABLE 3  
BULK SPECIFIC GRAVITY OF ROCK CORES

Core	Bulk Specific Gravity by		
	ASTM Method	Geometrical Mensuration Method	Mercury Displacement Method
1-Menlo	2.637	2.728	2.710
2-Pints	2.271	2.337	2.312
3-Alden	2.510	2.586	2.527
4-Linwood	2.636	2.734	2.694
5-Cook	2.565	2.634	2.622
6-Keota	2.489	2.592	2.549

RESULTS AND DISCUSSION  
OF INVESTIGATIONS

Rock Cores

The bulk specific gravity of rock cores was determined by ASTM Standard Method C 127, the geometrical mensuration method, and the mercury displacement method. Results are given in Table 3. It is evident from the data that the ASTM standard test gives a minimum value whereas the geometrical mensuration method gives a maximum value of bulk specific gravity. The values obtained by three methods have excellent correlations with each other, which indicates that consistent results have been obtained by using rock cores.

Plots are made of values obtained by the ASTM standard test and those obtained by geometrical mensuration in Figure 1. The correlation coefficient is 0.9969, which is significant at the 1 percent level. The equation of the regression line is

$$Y = -0.0735 + 1.0625X$$

where X = bulk specific gravity by the ASTM method, and  
Y = bulk specific gravity by geometrical mensuration.

Bulk specific gravity by geometrical mensuration is greater than that by the ASTM method. Differences range from 0.07 for light aggregate to 0.09 for heavy aggregate. A possible reason for this almost constant difference is the moist surface of the rock cores even after being rolled in absorbent cloth. Because of this, the saturated surface-dry weight observed is high, resulting in reduced bulk specific gravity. However, bulk specific gravity by geometrical measurements may give slightly higher results because of a slight undulation irregularity that is neglected in the measurement of dimensions. However, it seems that the moist surface plays a predominant role in causing this significant difference.

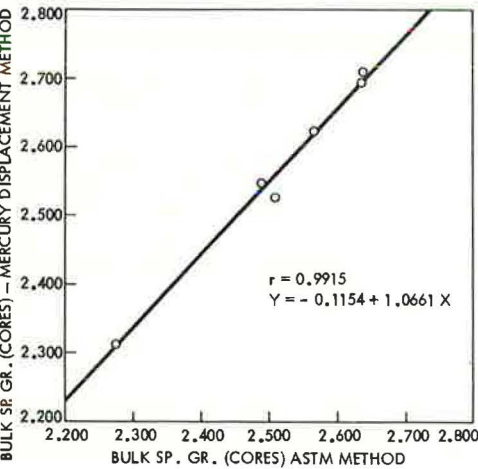


Figure 1. Bulk specific gravity of cores, ASTM method versus geometric mensuration method.

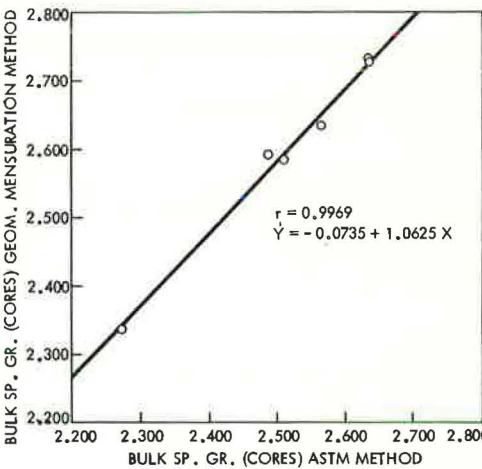


Figure 2. Bulk specific gravity of cores, ASTM method versus mercury displacement method.

The correlation coefficient for values obtained by the ASTM standard method and those by the mercury displacement method is 0.9915 (Fig. 2), which is significant at the 1 percent level. The equation for the regression line is

$$Y = -0.1154 + 1.0661X$$

where  $X$  = bulk specific gravity by the ASTM method, and

$Y$  = bulk specific gravity by the mercury displacement method.

Specific gravity obtained by the mercury displacement method is higher than that of the ASTM standard method. The difference ranges from 0.04 for light aggregate to 0.06 for heavy aggregate. Again, a possible reason is the moistness over the surface in the ASTM standard test.

From the results of these three methods, it thus appears that ASTM standard tests C 127 and C 128 may underestimate the bulk specific gravity as they take into account absorption as well as adsorption of the particles. This is indicated by the moistness that can be seen visually. This discrepancy had been pointed out by Shergold (8), who recommended that the aggregate should be exposed to the atmosphere for at least 10 minutes before being weighed after the aggregate has been wiped dry with absorbent cloth. However, this 10-minute time period cannot be adopted in all circumstances because of the varying air temperature and humidity conditions, but it does indicate the required improvement of the test.

Hughes and Bahramian (5) also obtained results showing that water absorption values obtained from the ASTM method were significantly higher than those obtained by their saturated air method. This further supports the underestimation of bulk specific gravity by standard test.

TABLE 4

BULK SPECIFIC GRAVITY OF COARSE AGGREGATES  
BY VARIOUS METHODS

Aggregate <sup>a</sup>	ASTM Method	CoCl <sub>2</sub> Method	FSS Method	Mercury Displacement Method
Cook				
1-S-B	2.567	2.601	2.615	2.560
1-L-A	2.613	—	—	2.556
1-L-B	2.603	—	—	2.526
Pints				
2-A	2.348	—	—	2.321
2-B	2.333	2.356	2.337	2.364
Alden				
3-S-B	2.475	2.537	2.487	2.496
3-L-A	2.517	—	—	2.546
3-L-B	2.508	—	—	2.555
Linwood				
4-A	2.613	—	—	2.644
4-B	2.582	2.620	2.631	2.658
Cook				
5-S-B	2.397	2.445	2.437	2.480
5-L-A	2.426	—	—	2.447
5-L-B	2.408	—	—	2.481
Keota				
6-A	2.326	—	—	2.322
6-B	2.263	2.270	2.250	2.257
Slag				
A	2.668	—	—	—
B	2.656	2.639	2.654	—
Trap				
A	2.822	—	—	—
B	2.857	2.900	2.903	—
Synopal				
B	2.149	2.180	—	—

<sup>a</sup>A = passing 1 in. sieve and retained on 3/8 in. sieve. B = passing 3/8 in. sieve and retained on No. 4 sieve.

## COARSE AGGREGATES

Bulk specific gravity of coarse aggregates was determined by four methods: ASTM Standard Method C 127, chemical indicator method using cobalt chloride, chemical indicator method using fluorescein sodium salt, and mercury displacement method. Average results based on duplicate determinations are given in Table 4.

Bulk specific gravity values determined by the ASTM method have been plotted in Figure 3 against those obtained by the mercury displacement method for 15 coarse aggregates. Correlation is very good—value of the correlation coefficient is 0.9924, which is significant at the 1 percent level. The equation of the regression line is

$$Y = 0.1738 + 0.9358X$$

where  $X$  = bulk specific gravity by the ASTM method, and

$Y$  = bulk specific gravity by the mercury displacement method.

As in cores, bulk specific gravity values obtained by mercury displacement are higher than those obtained by the ASTM method.

Values of bulk specific gravity obtained by using cobalt chloride (Table 3) as the indicator of saturated surface-dry condition

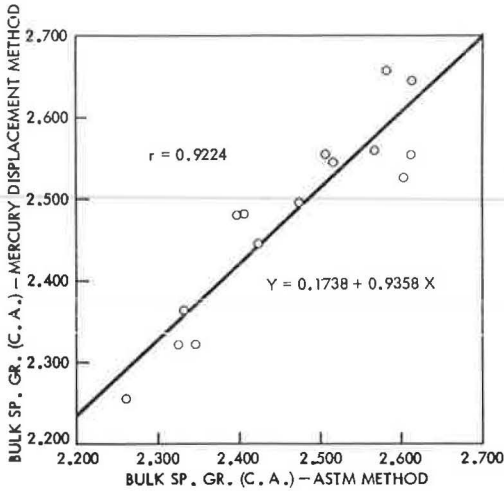


Figure 3. Bulk specific gravity of coarse aggregates, ASTM method versus mercury displacement method.

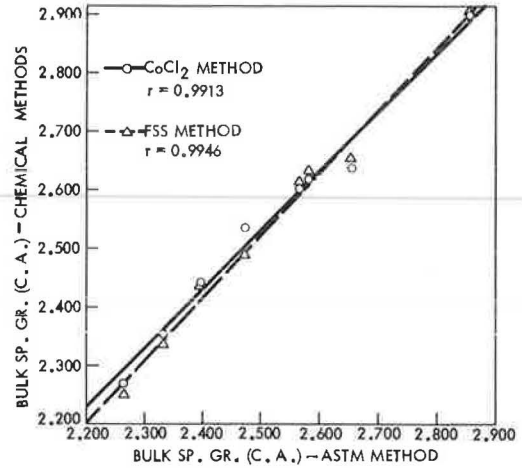


Figure 4. Bulk specific gravity of coarse aggregates, ASTM method versus gravity chemical dye method.

are higher than those obtained by the ASTM method. The difference is about 0.03 for the entire range of aggregates. Linear correlation is excellent (Fig. 4)—the correlation coefficient is 0.9913, which is significant at the 1 percent level.

The striking constancy of difference in the entire range of light to heavy aggregates, except slag, seems to point out that the chemical method takes care of the adsorbed water on the surface of the aggregates, because only anhydrous  $\text{CoCl}_2$  can have a blue color while  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  is red. Because of the disappearance of adsorbed water, the weight of saturated surface-dry aggregate in air is less, which gives an increased value of bulk specific gravity. It seems that the chemical method gives realistic bulk specific gravity values. It is further supported when these values obtained from the  $\text{CoCl}_2$  method are compared with those obtained by the mercury displacement method (Fig. 5). Correlation is excellent—the value of the correlation coefficient is 0.9671, which is significant at the 1 percent level. The discrepancy in values of bulk specific gravity in the case of slag obtained by the ASTM and chemical methods can probably be attributed to the cellular nature of slags, which retain internal water not held by capillary forces.

As in the case of cobalt chloride, fluorescein sodium salt has also indicated the saturated surface-dry condition of the aggregates fairly well. The values of bulk specific gravity obtained by using this indicator are shown in Figure 4 against those obtained by the ASTM standard method. The correlation coefficient is 0.9946, which is significant at the 1 percent level. The values obtained from the fluorescein sodium salt (FSS) method also have excellent linear correlation with those obtained

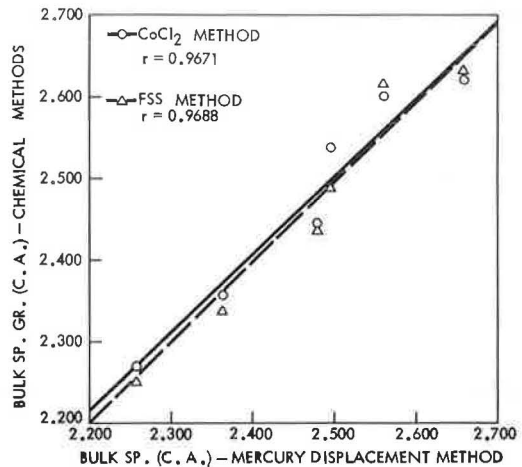


Figure 5. Bulk specific gravity of coarse aggregates, mercury displacement method versus chemical dye method.

TABLE 5  
BULK SPECIFIC GRAVITY OF FINE AGGREGATES

Aggregate	ASTM Method	CoCl <sub>2</sub> Method	FSS Method
Cook	2.551	2.553	2.535
Pints	2.284	2.288	2.295
Alden	2.498	2.480	2.492
Linwood	2.534	2.516	2.536
Cook	2.395	2.420	2.408
Keota	2.426	2.409	2.401

by mercury displacement. The linear correlation coefficient is 0.9688, which is significant at the 1 percent level. Comparison of the two chemical methods (Fig. 4) shows that the two indicators give practically identical results for an average weight aggregate.

### Fine Aggregates

Results of bulk specific gravity tests on fine aggregates (average value based on duplicate determinations) are given in Table 5. Figure 6 shows the linear correlations of ASTM bulk specific gravity with values obtained by using the two chemical methods.

With cobalt chloride, the correlation coefficient is 0.9862, which is significant at the 1 percent level, and the equation for the regression line is

$$Y = 0.1831 + 0.9237X$$

where  $X$  = bulk specific gravity by the ASTM method, and

$Y$  = bulk specific gravity by the CoCl<sub>2</sub> method.

As in the case of coarse aggregates, values obtained by the CoCl<sub>2</sub> method are higher than those obtained by the ASTM method. The difference is about 0.01 for the entire range of aggregates and is smaller than the 0.03 observed in the case of coarse aggregates.

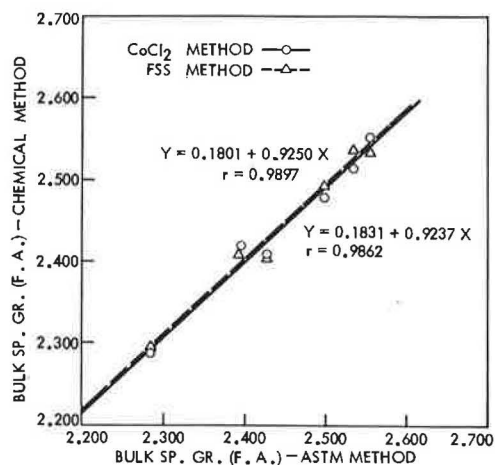


Figure 6. Bulk specific gravity of fine aggregates, ASTM method versus chemical dye method.

TABLE 6  
DIFFERENCES BETWEEN DUPLICATE DETERMINATIONS OF BULK SPECIFIC GRAVITY OF COARSE AGGREGATES

Coarse Aggregate	ASTM Method	CoCl <sub>2</sub> Method	FSS Method
Menlo	0.022	0.005	0.008
Pints	0.001	0.005	0.003
Alden	0.005	0.006	0.004
Linwood	0.022	0.008	0.011
Cook	0.006	0.003	0.007
Keota	0.019	0.009	0.012
Trap	0.014	0.004	0.019
Slag	0.020	0.022	0.015
Average difference	0.014	0.008	0.010
Range	0.001 to 0.022	0.003 to 0.022	0.003 to 0.019
Average difference, excluding slag	0.012	0.006	0.009
Range, excluding slag	0.001 to 0.022	0.003 to 0.009	0.003 to 0.019



With fluorescein sodium salt, the correlation coefficient is 0.9897, which is significant at the 1 percent level. The equation for the regression line is

$$Y = 0.1801 + 0.9250X$$

where  $X$  = bulk specific gravity by the ASTM method, and

$Y$  = bulk specific gravity by the FSS method.

The relationship is similar to that observed in the case of cobalt chloride.

### Repeatability and Reproducibility

**Coarse Aggregate**—Table 6 gives the differences between duplicate determinations for each coarse aggregate for three methods, that is, the ASTM standard method and the two chemical methods. It is evident from Table 6 that probably both the ASTM method and the chemical methods are not suited for determination of bulk specific gravity of slag.

From the data in Table 6 obtained from limited study, it appears that both the  $\text{CoCl}_2$  method and the FSS method give better duplicate results than the ASTM standard test. The  $\text{CoCl}_2$  method in particular gives duplicate determinations that check within 0.01, compared with the 0.02 provided in the case of the ASTM standard test, and is recommended for further investigations to establish its repeatability and reproducibility.

To compare the reproducibility of the standard ASTM method and the proposed  $\text{CoCl}_2$  method of bulk specific gravity determination, experiments were conducted on 6 materials involving three experienced operators. Duplicate determinations were made on all materials by all operators and with both methods. The results are given in Table 7. Average values based on the duplicate determinations have been calculated. Between operators, the maximum difference observed in value of bulk specific gravity by the  $\text{CoCl}_2$  method is 0.006, whereas for the ASTM method it is 0.026 (Table 7). Average variation between operators is 0.005 in the case of the  $\text{CoCl}_2$  method and 0.019 for the ASTM standard test. In all cases, bulk specific gravity by the ASTM method is less than that by the  $\text{CoCl}_2$  method. The difference is greater, in the case of absorptive aggregates (2-B and 3-S-B).

**Fine Aggregate**—Table 8 gives the differences between duplicate determinations for each fine aggregate for the ASTM standard method and the two chemical methods. The data given in Table 8 also indicate the better performance of chemical methods, which

TABLE 7  
RESULTS OF REPRODUCIBILITY STUDIES FOR COARSE AGGREGATES,  
VALUES OF BULK SPECIFIC GRAVITY

Aggregate	Operator L	Operator D	Operator K	Maximum Variation Between Operators
1-S-B				
$\text{CoCl}_2$	2.615 > 2.608 2.600	2.608 > 2.604 2.599	2.605 > 2.602 2.598	0.006
ASTM	2.576 > 2.577 2.577	2.598 > 2.589 2.588	2.590 > 2.590 2.592	0.013
2-B				
$\text{CoCl}_2$	2.404 > 2.411 2.417	2.402 > 2.409 2.415	2.401 > 2.408 2.414	0.003
ASTM	2.318 > 2.318 2.317	2.334 > 2.337 2.339	2.335 > 2.333 2.331	0.019
3-S-B				
$\text{CoCl}_2$	2.529 > 2.526 2.522	2.530 > 2.527 2.524	2.518 > 2.521 2.523	0.006
ASTM	2.446 > 2.449 2.452	2.475 > 2.475 2.474	2.472 > 2.474 2.475	0.026
5-S-B				
$\text{CoCl}_2$	2.554 > 2.552 2.550	2.560 > 2.556 2.552	Not done	0.004
ASTM	—	—	—	—



TABLE 8  
DIFFERENCES BETWEEN DUPLICATE DETERMINATIONS OF BULK  
SPECIFIC GRAVITY OF FINE AGGREGATES

Fine Aggregate	Difference		
	ASTM Method	CoCl <sub>2</sub> Method	FSS Method
Menlo	0.014	0.007	0.002
Pints	0.000	0.008	0.001
Alden	0.004	0.009	0.002
Linwood	0.007	0.014	0.013
Cook	0.008	0.005	0.006
Keota	0.021	0.004	0.008
Average difference	0.009	0.008	0.005
Range	0.000 to 0.021	0.004 to 0.014	0.001 to 0.013

TABLE 9  
RESULTS OF REPRODUCIBILITY STUDIES FOR FINE AGGREGATES,  
VALUES OF BULK SPECIFIC GRAVITY

Aggregate	Operator L	Operator D	Operator K	Maximum Variation Between Operators
1-S-C				
CoCl <sub>2</sub>	2.574 > 2.574 2.573	2.637 > 2.636 2.634	2.552 > 2.552 2.551	0.084
ASTM	2.571 > 2.566 2.561	2.569 > 2.569 2.568	2.560 > 2.559 2.557	0.010
3-S-C				
CoCl <sub>2</sub>	2.466 > 2.465 2.464	2.522 > 2.521 2.519	2.510 > 2.509 2.507	0.056
ASTM	2.481 > 2.484 2.486	2.553 > 2.548 2.542	2.491 > 2.489 2.486	0.064

may give duplicate determinations checking within 0.01. Because CoCl<sub>2</sub> is very inexpensive in comparison with fluorescein sodium salt, the former is preferred.

Between operators, the maximum difference observed in value of bulk specific gravity by the CoCl<sub>2</sub> method is 0.084, whereas for the ASTM method it is 0.064 (Table 9). It seems that overdrying occurred in some of the tests, resulting in higher bulk specific gravity.

#### SUMMARY AND CONCLUSIONS

The following conclusions can be drawn from this study, wherein a new method has been proposed:

1. Investigations have shown that the ASTM standard tests underestimate the bulk specific gravity of aggregates because these tests measure adsorption as well as absorption of particles. Results obtained in determination of 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 in between those obtained from the ASTM standard method and the geometrical mensuration method. Further investigations are needed to establish a properly specified pressure at which the 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 because color change is quite apparent.

4. Chemical methods achieve the elimination of the moistness over the aggregates and thus tend to give realistic values of bulk specific gravity. Attainment of saturated surface-dry condition in the case of fine aggregates by the chemical method is not affected by the interlocking of the particles.

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

6. The cobalt chloride method in general appears to be the preferable of the two chemical methods investigated, considering the data in this study and the relative cost. However, fluorescein 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 compared with the 0.02 specified in the ASTM standard test, although extensive tests should be made in various laboratories to establish the reproducibility and limitations of this method.

#### ACKNOWLEDGMENTS

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### *Appendix*

#### DETERMINATION OF BULK SPECIFIC GRAVITY OF AGGREGATES BY CHEMICAL INDICATOR METHODS

##### Cobalt Chloride Method

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

1. The sample is immersed in a 5 percent solution of cobalt chloride instead of plain water for 24 hours.
2. The sample is removed from the solution and put on a table, which should have a white top, or it can be put in a wide, white enameled tray. The aggregate may not appear to be colored, but the visible water films on the aggregate will be pinkish in color.

3. The sample is spread out 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 a bluish color. The aggregate should be turned over frequently by gentle hand action. If there is still some moisture 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 the saturated surface-dry condition.

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

#### Fluorescein Sodium Salt Method

The procedure is essentially the same as the cobalt chloride method. In this case a 0.5 percent solution of fluorescein sodium salt is used. The sample is dried in the same manner as in the cobalt chloride test. The sample acquires a yellowish color when taken from the solution after 24 hours of immersion. On drying, an orange color appears on the aggregates; the porcelain also changes to a distinct orange. This is assumed to be the saturated surface-dry condition, and the sample is further treated according to the ASTM C 127 and C 128 test procedures.