*THE INFLUENCE OF FINE AGGREGATE
GRADATION CHARACTERISTICS
ON THE AIR ENTRAINMENT
IN PORTLAND CEMENT MORTAR*

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by
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PURDUE UNIVERSITY
LAFAYETTE INDIANA

Final Report

THE INFLUENCE OF FINE AGGREGATE GRADATION CHARACTERISTICS ON AIR ENTRAINMENT IN PORTLAND CEMENT MORTAR

To: G. A. Leonards, Director
Joint Highway Research Project

May 10, 1966

From: H. L. Michael, Associate Director
Joint Highway Research Project

File: 5-14-3
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Attached is a paper entitled "The Influence of Fine Aggregate Gradation Characteristics on Air Entrainment in Portland Cement Mortar" by D. W. Deno. This was Mr. Deno's thesis for the MSCE degree. His major Professor was Dr. C. F. Scholer. Mr. Deno is now employed at the Portland Cement Association research laboratory in Skokie, Illinois.

Respectfully submitted,

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D. W. Deno

Joint Highway Research Project

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ABSTRACT

Deno, Douglas Willard. M.S.C.E., Purdue University, June 1966.

The Influence of Fine Aggregate Gradation Characteristics on Air Entrainment in Portland Cement Mortar. Major Professor: Charles F. Scholer.

This investigation was undertaken to determine how certain fine aggregate gradation characteristics influence air entrainment in portland cement mortar. A local sand of glacial origin was used for most of the work although a quartzite sand was used in one instance for comparison purposes.

A number of separate investigations were conducted. One of these was concerned with the entraining ability of different mixer paddles. It was found that different paddles do entrain differing amounts of air.

It was found that air content varies with changes in both fineness modulus and specific surface, with fineness modulus having the most predominant influence. However, fineness modulus and specific surface were found not to be enough gradation control to predict air contents.

The two sands investigated gave similar, but not identical results. Thus, air entrainment may also be influenced by mineralogical composition, aggregate shape or other aggregate characteristics.

An investigation of single size fractions of sand showed that size fractions No. 8 - No. 16 and No. 50 - No. 100 entrain similar amounts of air and this amount is less than that entrained by size fractions No. 16 - No. 30 and No. 30 - No. 50.

A good relationship was found between air content and the amount of No. 8 - No. 16 sand present in the aggregate gradation. A poor relationship was found between air content and the amount of No. 30 - No. 50 present.

INTRODUCTION

Entrained air induces a number of desirable properties in concrete. It increases the workability of fresh concrete, thus allowing the use of less water in the mix. It also reduces the tendency of concrete towards segregation and bleeding. Hardened concrete containing entrained air is less susceptible to the disrupting effects of freezing and thawing action, and this is the principal reason for the use of air entrainment. It is also more resistant to sulfate action and to the action of de-icing salts. Air-entrained concrete has a better degree of water-tightness than does non-air-entrained concrete.

However, the advantages of entrained air can be achieved only if the proper amount of air is entrained in the concrete. An insufficient amount of entrained air will not give the concrete the desired degree of durability. If too much air is entrained, the result will be a concrete of lower strength than anticipated.

A recent study (1)* indicated that bridge decks which deteriorated were usually constructed of concrete which contained insufficient amounts of entrained air. Another investigation (2) indicates that in some instances the proper amount of air is not being entrained in concrete used for paving purposes. Data obtained during the latter investigation also indicated a wide variation in the amount of air entrained in supposedly similar batches of concrete. It was found that the mean air

* Numbers in parentheses refer to references listed in the bibliography.

contents of the mixes sampled were in the lower range of the specified limits of four to seven percent air (3). This is not good, since, if the average air content is low, say about 4.5 percent, and the standard deviation is large, say about 0.5 percent, then a significant number of batches would have air contents below the specified minimum of four percent.

Since, within limits, the amount of air entrained in a given concrete depends primarily on the amount of air-entraining agent used, it seems logical that the best possible solution to the problem of low air contents would be an increase in the amount of agent used. However, sometimes extra dosages of the agent do not appreciably affect the air content after it reaches a certain level. Neville (4) states that "there is a maximum amount of any agent beyond which there is no increase in the volume of voids."

The amount of air entraining agent is not the only factor influencing the amount of air that can be entrained in concrete. There are a large number of other variables which are involved in this problem: type of air-entraining agent, type of cement, water-cement ratio, aggregate-cement ratio, type of mixing action, mixing temperature, mixing time, consistency of concrete, amount of vibration during placing, finishing methods and a large number of aggregate characteristics. The sand, or fine aggregate, portion of a concrete aggregate seems to be that part of the aggregate which has the greatest influence on air entrainment.

The amount of air entrained in a concrete can be controlled to a certain extent by the amount of sand in the mix. An increase in the amount of sand will usually result in an increase in the amount of air and vice versa.

A number of researchers have found that the grading characteristics of sand have an effect on the amount of air entrained in concrete (5, 6, 7, 8). One article contained the following statement: "Field reports indicated difficulty with certain sands in securing the desired amount of entrained air; this was frequently attributed to the size grading of the sand, particularly to a deficiency in the finer fractions" (8). Other articles contain similar statements suggesting that the fine aggregate gradation characteristics do affect air content.

Air content has been found to vary with fineness modulus, which is a non-dimensional measurement of the average size of a sand. However, fineness modulus is somewhat sensitive to small changes in the amounts of the coarser size fractions of the sand. Another gradation characteristic which is also a measurement of particle size is specific surface. This is a measurement of surface area per unit weight. Specific surface is very sensitive to small changes in the amount of the finer fractions of the sand. A limited amount of work has been done concerning air entrainment as influenced by specific surface, so perhaps more work should be done in this area. At the present time, almost all concrete aggregate specifications contain a requirement for fineness modulus, and few, if any, contain a statement pertaining to specific surface. Perhaps the two can be combined to give a more precise meaning to fine aggregate gradation.

Different size fractions of sand entrain different amounts of air when taken separately. However, there has been some disagreement as to which size fractions of the fine aggregate have the greatest influence on air entrainment in concrete. It is generally agreed that the fine aggregate fractions retained on the No. 16 sieve and passing the No. 100

sieve have little or no influence on the amount of air entrained in a concrete mix, but there is no overall agreement as to which of the intermediate size fractions has the most pronounced effect. It is possible that the amount of particular size fractions present in the gradation may be very significant in entraining air, while the amount of other size fractions has no influence on air entrainment.

REVIEW OF LITERATURE

Entrained air has been used in concrete as a means to increase its resistance to frost action since the late 1930's. It has since been recognized that there are other benefits to be gained when air is entrained in concrete.* It has also been realized that the control of the air content of a concrete mix is no small problem. There are numerous factors which influence air entrainment. The problems involved with and the effects of these factors are not completely resolved and understood.

One important factor which influences the amount of air entrained in concrete is the gradation of the aggregate used in the mix (4, 5, 6, 7, 8, 9, 10, 11, 12). Jackson (9) wrote in 1944: "Test data and experience both show that the quantity of air entraining agent which will be required for optimum air content for any given combination of conditions will vary considerably, depending upon a number of factors, including ... character and grading of aggregate" At about the same time Chubb (10) made an almost identical statement, as did Cordon (11) in 1946 in an article discussing entrained air as a factor in concrete mix design. Singh (5) stated that the grading of the aggregate is "among the factors of greatest practical importance" in the problem of control of air entrainment. A paper published by the Portland Cement Association (12)

* These other benefits were enumerated on Page 1 and will not be discussed here because there is an abundant amount of literature available on this subject.

states that: "Variations in air content can be expected with variations in aggregate proportions and gradation, mixing time, temperature, and slump."

The fine aggregate, or sand portion, of concrete aggregate has been shown to have the most effect on air entrainment. The gradation of the coarse aggregate fraction has little or no effect on the amount of air entrained. Many investigations of aggregate gradation and air entrainment have been concerned with the fine aggregate (6, 8, 13, 14, 15). Walker and Eloem (13) make this statement: "Only a few tests have been made on the effect of grading of coarse aggregate, but the limited data available indicate that the coarse aggregate has little effect, except in so far as it affects the amount of sand required." Craven (6) stated that "air entrainment increased with the proportion of sand used in a mix ...", and Powers (15) said that the air content "varied with the grading of the aggregate, especially with the sand grading and the percent sand in the aggregate."

It is agreed upon in general that an increase in the amount of fine aggregate in a mix will result in an increase in the air content. The Portland Cement Association's paper mentioned above (12) states that "increasing the amount of fine aggregate causes more air to be entrained for a given amount of air-entraining cement or admixture." However, the resulting increase in air content due to an increase in the sand fraction of the aggregate may not be a beneficial type of increase. A paper (16) by a subcommittee of the Highway Research Board Committee on Chemical Additions and Admixtures for Concrete contains the following remarks: "Increasing the amount of sand will increase the amount of air. However, the spacing factor is probably not greatly affected. It is

therefore incorrect to attempt to increase the air content by increasing the sand proportion, especially considering the increased water demand such a change would involve."

Another method of changing the air content is by a change in the sand gradation rather than a change in the amount of sand. Scripture, Hornibrook and Bryant (8) support this idea with the following remark: "Another factor which it is commonly believed has an important influence on air entrainment is the size grading of the sand used in the concrete mix." Walker and Eloem (13) stated that: "the amount of air entrained was significantly affected by the grading of the fine aggregate"

There is agreement on the fact that within the fine aggregate the middle and coarser fractions allow more air to be entrained than the finer fractions. Singh (5) says that "as regards the grading of the aggregates ... as the sand becomes coarser, more air is entrained when all other factors are equal." Thomson (17) makes this statement: "The amount of entrainment with a given amount of agent or admix decreases with an increase of fines in the sand aggregate."

Most investigators who have considered the effect of fine aggregate gradation on air entrainment have studied the effect of the various size fractions. Mortars were mixed using only one size fraction of the aggregate, and the amount of air entrained was determined. The size fraction which entrained the most air was then singled out as that size fraction having the most influence on air entrainment. There is disagreement, however, as to the specific fraction which entrains the most air. The safer statement would probably be one which includes two or three of the sand fractions. The Portland Cement Association paper (12) says that "fine aggregate particles in the middle sizes result in more air than the

very fine or coarse sizes." Troxell and Davis (14) state that "the entrainment appears to take place most effectively in the sand constituent between the size range represented by the No. 100 to the No. 14 sieves. In fact, experiments have shown that mixtures with sufficient sand in this range, but with no cement at all, can be made plastic by air entrainment." Others make statements that single out a particular sand size (4, 5, 6, 8, 13, 18, 19, 20). In a test program conducted by Scripture, Hornibrook and Bryant (8) it was found that "with all three air-entraining agents and also in the mixes without air entraining agents, a maximum amount of air is entrained by using the 14-28 mesh size." Walker and Bloem (13) make this statement: "The amount of air entrained was significantly affected by the grading of the fine aggregate and appeared to be principally a direct function of the amount of the No. 50 to No. 30 grain size." Craven (6) also made a similar statement. Mielenz, Wolkodoff, Eackstrom and Flack (18) support this idea and present an explanation of why the No. 30 to No. 50 fraction has the most influence on air entrainment. They wrote:

"... air content of concrete is increased by increase in the proportion of sand of intermediate size, particularly in the ranges passing the No. 30 and retained on the No. 50 or the No. 100* sieves, and is decreased by increase in coarser or finer sizes of aggregate or of cement. This size fraction is critically important because the openings between grains in this size range are small enough to impede movement of portland cement, yet the grains are large enough to retain enmeshed among their

* The precise range found to be most effective will vary with the characteristics of each fine aggregate, particularly with respect to grading within this range, particle shape, surface texture, and mineralogic composition.

granular framework bubbles big enough to withstand rapid dissolution in the mixing water in the presence of a satisfactory air-entraining agent. In a granular mass composed of spherical particles of uniform size in the most compact arrangement, the diameter of the largest sphere that can be contained in the interstitial space is 0.22 times the particle diameter. Lack of uniform size and departure from sphericity will decrease the size of the enclosed space if the most compact arrangement is achieved.

"The maximum size of the space subtended by particles passing the No. 30 and retained on the No. 100 sieve varies from about 33 to 130 microns. As will be shown in subsequent sections, 90 to 99 percent of the air voids in air-entrained concrete are less than 100 microns in diameter and so can be contained among sand particles in this size range. Moreover, it will be shown that bubbles larger than about 60 microns are far more susceptible to escape from the concrete during vibration than are smaller ones, there being a sharp change in the rate of loss at about this size. An increase in the proportion of sand finer than that which is optimum for entraining air will decrease the available volume among the particles in this fraction and will consequently subject the displaced bubbles to greater increased pressure, thus facilitating their escape from the concrete and increasing the solubility of the air and hastening their dissolution. An increase in coarser aggregate decreases the available interstitial space of optimum dimensions because a portion of the finer sand is replaced by aggregate particles.

"Hence, it appears that clusters of grains in approximately the No. 30-50 or No. 30-100 size range are an important reservoir for those bubbles which persist until hardening of the concrete."

In a discussion of particular size fractions Singh (5) makes the following remarks: " ... it is thought that entrained air increases with the amount of material passing the No. 30 and retained on the No. 50 sieve. Though it is not disputed that the various size groups play some part in entraining air, indication of their relative importance is still somewhat lacking." In a discussion of his test results, Singh says: "Examination of the amount of gross (or naturally) entrained air in relation to the amount of 25-52 size group for each grading indicates wide scatter, though there is a general tendency for the percentage of air to increase with the amount of this size. A better relationship is, however, obtained between the percentage of 52-100 size group and the amount of gross entrained air. As the amount of material of this size increases, there is a marked tendency for the air to decrease. It is ... quite clear that particles of this size group are not so conducive to air entrainment."

Kennedy (19), in 1944, investigated the effect of various size fractions of sand. He said: " ... our investigation of the sand constituent showed that air entraining agents were not effective on size particles larger than retained on 14 mesh. Hence a study of the various particle sizes of sand finer than No. 14 in the presence of air-entraining agents was conducted. The average relative amount of air entrained by the various sand fractions using identical mixing procedures, were as follows:

<u>Size</u>	<u>Percent Air</u>
14 - 28	15 - 20
28 - 48	30 - 35
48 - 100	45 - 50
- 100	0 - 1

It is obvious that radical changes in sand gradation could alter materially the total air entrained in a concrete."

Singh (5), in referring to particles passing the No. 100 sieve, said that indications show "that their effect will most probably be negligible under practical conditions."

An article in the Concrete Industries Yearbook (20) contained this statement: "An important fact to remember is that a part of the mixing action is influenced by the grading of the sand. The size portions of sand that are denoted by the No. 30 and No. 50 sieves help most to entrain air. The finer, No. 100 sieve portion helps create a fatty, cohesive mix which holds the air that has been entrained; while the minus No. 100 fraction tends to soften the mixing action, reduces foaming and inhibits air entrainment. This air-inhibiting quality pertains to excessive quantities of any very fine material in the mix. Often sudden major variations in air content can be traced to changes in the percentages of materials in these size ranges."

Scripture, Hornibrook and Bryant (8) state that the 50-100 and minus 100 fractions contribute very little to air entrainment: "In mortar mixes effects are observed similar to those obtained using sand and water without cement, but the differences with size grading are less marked. It also appears that the point at which maximum air entrainment is secured moves toward the coarser sizes. It is fairly definitely shown that the sizes 48-100 mesh and minus 100 mesh contribute very little to air entrainment."

Powers (7) discusses the idea that specific size fractions alone control the amount of air in a mix with the following statements:

"... some investigators concluded that in aggregate alone, or in concrete,

each size group has a specific air-entraining ability. But it now seems clear that no one size of particle nor one size-group can have an independent effect in a mixture comprising many sizes. As we have seen, the aggregate provides a screen that holds bubbles when the cement is absent, or that holds bubbles in plastic concrete when no air-entraining agent is used: and its voids, slightly dilated, provide the space for paste and bubbles. Therefore, the relationship between air content of a given mixture and aggregate characteristics is determined by whether or not emulsification is the major entraining process, as well as by aggregate void content in the dilated state, and sometimes by void size. The latter two factors are functions of size range and grading; for a given size range, aggregate characteristics vary with grading. If grading of aggregate for a given mix is changed by increasing the proportion of a given size group, a change in air content of the mixture may be obtained, and thus the change in air content seems to be the effect of the particular size group that was increased. But the effect actually is due to change in void content of the aggregate, and possibly to change in void size."

There has been some comment on the relationship between the fineness modulus of the sand and the amount of air entrained. Walker and Bloem (13) state that: "A good relationship was found between fineness modulus and amount of air entrained. The quantity of air increased from the lowest fineness modulus to a peak at about 2.5 and, thereafter, decreased sharply. These data are not shown since other studies suggest that the relationship is not general and that it would differ for other gradings of the same fineness moduli."

Craven (6) said that: "Generally the percentage of air entrained in concrete increased with decrease in fineness modulus of sand irrespective

of the agent used and these results were similar when no agent was used."

Singh (5, 21, 22, 23) has done much work on the effect of specific surface of aggregates on the properties of concrete. He states that "aggregate gradings of the same specific surface, for practical purposes, have the same concrete making properties" (23). However, in a study of the effect of aggregate grading on air entrainment (5), he found that aggregate gradings of the same specific surface gave concretes with different air contents.

Singh discusses the problem of determining the specific surface of aggregates (21) and refers to work done on the subject by Loudon (24): "Loudon has tested a few British sands and expressed their angularity as a ratio of the specific surface of a size group to the specific surface of spheres of a corresponding size group." ... "He suggests $f^* = 1.1$ for rounded sand, $f = 1.25$ for sand of medium angularity, and $f = 1.4$ for angular sand." Singh feels that specific surface, as a measure of average particle size, is a good and useful way of stating this characteristic of an aggregate gradation.

A subject that has been the cause of much debate when air content is discussed is that of "entrapped" air versus "entrained" air. Powers (7) feels that when an air-entraining agent is used, the resulting air volume is all entrained air. He says: "The air content of concrete containing intentionally entrained air is considered by some observers to comprise two categories: entrained air and entrapped air. Entrapped air is usually taken to be that which is found in a given mixture when an air-entraining agent is not used, and accordingly, entrained air is

* f = angularity factor.

sometimes said to be that in excess of entrapped air, present because of the air-entraining agent. Such distinctions are doubtful as to accuracy and utility. All air that is an ingredient of the mixture (which distinction excludes air in voids produced by segregation of coarse material or by incomplete filling of forms by a stiff mixture) is held by the same mechanisms, and when an air-entraining agent is present we may safely assume that all the bubbles carry films due to the agent, whether the bubbles appear spherical or not." He also states that "the total air content obtained in concrete by a given dosage of air-entraining agent in a given mix depends under most circumstances on the amount of air that would be present if the agent were not used." ... "The amount of air added by means of a given dosage tends to be independent of factors that determine the air content without agent, provided that consistency is a constant factor."

Neville (4) says that: "The volume of air entrained in a given concrete is independent of the volume of accidental air and depends primarily on the amount of air-entraining agent added." However, Singh (5) came to the conclusion that "the gross entrained air is related to the air entrained under natural conditions; that is, an aggregate that will entrain a large amount of air under natural conditions will entrain proportionally more air with an air-entraining admixture." The HRB paper (16) previously mentioned contains this discussion: "Ordinary concrete contains about one or two percent air in the form of relatively large voids. This is termed entrapped air. Entrained air, on the other hand, exists as very small bubbles and in an amount that depends on the amount of the air-entraining agent added to the concrete as well as upon other variables of the mixture and environment."

The human element is a factor which cannot be overlooked when discussing the control of air entrainment. Timms (25) supports this with the following remarks: "The results obtained with 25-year-old air-entrained concrete have proved very satisfactory, indeed. The few projects in which performance was not satisfactory could usually be traced to poor judgment in the selection of the percentage of air required or poor control of the materials, so that the required air content was not in the concrete at the time of placing." All discussion of this nature can be pretty well summed up in these remarks of Waddell (26): "Air entrainment is not a cure-all for whatever distress concrete may suffer. Entrained air does improve the durability and other characteristics of concrete, and its use should not be undervalued. But it cannot take the place of good materials and competent workmanship. These comprise the foundation of sound concrete construction."

PURPOSE AND SCOPE

This study is concerned with the effect that variations in aggregate gradation characteristics have on the amount of air entrained in portland cement mortar.

The purpose of this study was 1) to determine how air content is affected by varying the specific surface while maintaining various levels of fineness modulus, 2) to determine how air content is affected by varying the amount of material of particular size fractions of sand present in the aggregate gradation while maintaining various levels of fineness modulus, 3) to determine if the observed effects were due to gradation differences or if they might be due to some other aggregate characteristics and 4) to see whether different mortar mixers would entrain relatively similar amounts of air in batches of mortar made with identical aggregate gradations.

This study was limited to mortar because the primary interest was with fine aggregate. It must be remembered, therefore, that the effects observed will be of a smaller magnitude when applied to concrete.

The investigation was confined to two different sources of fine aggregate.

MATERIALS AND EQUIPMENT

Materials

The fine aggregate used for most of the mixes in this study was a local sand obtained from a river terrace deposit of glacial origin. This fine aggregate is designated 79-1G sand in the Joint Highway Research Project concrete laboratory at Purdue University and will hereafter be referred to by that designation.

A sieve analysis was performed on the 79-1G sand in accordance with ASTM C 136-63, Standard Method of Test for Sieve or Screen Analysis of Fine and Coarse Aggregates and the specific gravities and absorption were determined in accordance with ASTM C 128-59, Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate. The results of these tests are presented in Table 1. The specific surface was determined for each aggregate fraction and these values are also shown in Table 1. A detailed procedure of the method of determining the specific surfaces and the corresponding data are given in Appendix A.

A quartzite sand was used for making a series of comparison batches of mortar. This sand, known as the Rib Hill Quartzite, is Precambrian in age and is one of the metamorphic sedimentary rocks of the Huronian Formation. It is quarried and crushed near Wausau, Wisconsin. The chemical analysis and physical properties of this sand are listed in Table 2.

TABLE 1
SIEVE ANALYSIS AND PHYSICAL PROPERTIES
OF 79-1G SAND

<u>Sieve Analysis and Specific Surface</u>			
<u>Passing Sieve No.</u>	<u>Retaining Sieve No.</u>	<u>Percent</u>	<u>Specific Surface (cm²/gm)</u>
3/8"	4	1	3.85
4	8	15	7.65
8	16	23	15.50
16	30	26	31.35
30	50	25	62.99
50	100	7	125.50
100	200	2	260.69
200	-	1	521.93

Physical Properties

Fineness Modulus = 3.08

Bulk Specific Gravity = 2.58

Bulk Specific Gravity (Saturated Surface Dry) = 2.62

Apparent Specific Gravity = 2.67

Absorption = 1.53%

TABLE 2
CHEMICAL ANALYSIS AND PHYSICAL PROPERTIES
OF CRUSHED QUARTZITE SAND*

<u>Chemical Analysis</u>	
<u>Compound</u>	<u>Percent Present</u>
SiO_2	98.86
$\text{Al}_2\text{O}_3 + \text{TiO}_2$	0.79
Fe or Fe_2O_3	0.09
Na_2O_3	0.07
K_2O	0.19

Physical Properties

Apparent Specific Gravity = 2.65

Loss on Ignition at 1000°C = 0.14

Melting Point - Range of 3040°F to 3400°F .

Hardness, Moh's Scale = 7

Weight per cubic foot = 90 to 110 pounds, depending on size.

This sand is insoluble in acids and alkali, but can be fluxed with alkali by heat, and then the silicate will be soluble in water.

* Manufacturer's analysis.

All of the aggregates were sieved and separated into the following size fractions:

<u>Passing Sieve</u>	<u>Retained on Sieve</u>
No. 4	No. 8
No. 8	No. 16
No. 16	No. 30
No. 30	No. 50
No. 50	No. 100
No. 100	No. 200
No. 200	---

The aggregate was also washed to remove dust particles.

A Type I portland cement from a single clinker batch was used in all of the mixes. The physical and chemical properties of this cement are listed in Table 3.

The air entraining agent used was a neutralized vinsol-resin solution which met the specifications of ASTM C 260-63T, Tentative Specifications for Air-Entraining Admixtures for Concrete. Two gallons of this agent were obtained from a larger drum and were stored in air-tight containers in the laboratory.

The mixing water used was purified to 1 ppm impurities before usage.

TABLE 3

PHYSICAL AND CHEMICAL PROPERTIES OF CEMENT*

Physical Properties

Fineness, No. 325 sieve = 95.9 percent

Specific Surface, Blaine = 3380 sq. cm. per gm.

Initial Set - 3 hrs., 15 min.

Final Set - 5 hrs., 5 min.

Chemical Analysis

<u>Compound</u>	<u>Percent Present</u>
Silicon dioxide, SiO_2	21.76
Aluminum oxide, Al_2O_3	5.41
Ferric oxide, Fe_2O_3	1.97
Calcium oxide, CaO	65.30
Magnesium oxide, MgO	1.11
Sulphur trioxide, SO_3	2.43
Loss on Ignition	1.78

Calculated Compound Composition

<u>Compound</u>	<u>Percent Present</u>
Tricalcium silicate, C_3S	51.20
Dicalcium silicate, C_2S	23.83
Tricalcium Aluminate, C_3A	11.00
Tetracalcium Aluminoferrite, C_4AF	5.99
Calcium Sulphate, CaSO_4	4.13

* The portland cement is designated No. 317 in the Joint Highway Research Project concrete laboratory at Furdue University.

Equipment

The equipment used for material preparation, mixing and air content determinations were sieving machines, mixers, flow table, air meter and other miscellaneous laboratory equipment.

For separating the aggregate into the various fractions mentioned above the following equipment was used:

1. "Gilson" mechanical sieving machines.
2. "Ro-tap" testing sieve shakers.
3. U.S. Standard Sieves, sizes $3/8"$, No.'s 4, 8, 16, 30, 50, 100 and 200.

The mortar mixers used were "Hobart" mixers, one meeting ASTM C 305-59T, Tentative Method for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency, and the other being of a similar nature and capacity, but with a "beater" type paddle. One of the mixers is shown in Figure 1 with both paddles. Paddle 1 meets the ASTM specification mentioned above, and Paddle 2 does not.

A flow table meeting ASTM C 230-61T, Tentative Specifications for Flow Table for use in Tests of Hydraulic Cement was used for determining the consistency of the mortar.

The air contents of the mortars were determined by using a volumetric method based on the principle of ASTM C 173-58, Standard Method of Test for Air Content of Freshly Mixed Concrete by the Volumetric Method and the "Chace" air meter. The air meter used is shown in Figure 2. The bowl of this air meter has a capacity of approximately 50 cc. which is about fourteen times the capacity of the "Chace" meter bowl. The air meter was calibrated so as to enable one to determine the air content to the nearest 0.1 percent.

FIGURE 1. MECHANICAL MIXER AND TWO PADDLES
USED FOR INVESTIGATION NO. 1.

FIGURE 2. VOLUMETRIC AIR METER

PROCEDURE

Investigations

This study consisted of nine basic investigations. All of these consisted of mixing batches of mortar and determining the resulting air contents. The methods of mixing and determining the air contents are described in detail in later sections. The nine investigations are described independently.

Investigation No. 1

Preliminary Investigation of Mixer Interaction and the Effect of Fineness Modulus and Specific Surface on Air Entrainment. There were two reasons for this investigation. The first was that the "Hobart" mixer meeting ASTM specifications (Paddle No. 1, Figure 1) could not be used for any mix containing aggregate particles which would be retained on the No. 8 sieve. The other "Hobart" mixer (Paddle No. 2, Figure 1) could mix larger particles, but did not meet ASTM specifications. Thus, if it could be shown that the two mixers gave statistically identical results, then the second mixer could be used and thus mixes containing larger particles could also be used. As will be shown in a later section, a mixer interaction was present and therefore the mixer meeting ASTM specifications was used for all remaining investigations.

The second purpose was to determine if either fineness modulus or specific surface or both had any effect on air entrainment.

Twelve aggregate gradations were set up, having no particles retained on the No. 8 sieve. These are shown in Table 4. The gradations were chosen such that the experimental design was a factorial one between fineness modulus and specific surface. The fineness moduli were 2.40, 2.70 and 3.00, and the specific surfaces were 50, 55, 60 and 65 cm^2/gm . One batch of each gradation was made in each mixer and one air content determination taken for each batch. If the analysis of the results had shown a "borderline" case, then more batches would have been mixed. It turned out that no more batches were necessary. The order of mixing was set up by randomization as explained in Appendix B.

TABLE 4

AGGREGATE GRADATIONS FOR INVESTIGATION NO. 1

Aggregate Gradation Number	Fineness Modulus	Specific Surface (cm^2/gm .)	Percent Retained on Sieve Number					
			8	16	30	50	100	200
1	2.40	50	0	1	39	100	100	100
2	2.40	55	0	10	40	90	100	100
3	2.40	60	0	20	40	80	100	100
4	2.40	65	0	20	38	90	92	100
5	2.70	50	0	20	70	80	100	100
6	2.70	55	0	30	70	70	100	100
7	2.70	60	0	30	68	80	92	100
8	2.70	65	0	40	61	80	89	100
9	3.00	50	0	60	70	70	100	100
10	3.00	55	0	60	62	88	90	100
11	3.00	60	0	50	82	83	85	100
12	3.00	65	0	60	77	80	83	100

Investigation No. 2

Air Content of Standard Mortar. In this part of the investigation three batches of mortar were made and tested according to ASTM C 185-59, Standard Method of Test for Air Content of Hydraulic Cement Mortar, using the same concentration* of the vinsol-resin air-entraining agent as was used in all other investigations. The reason for this step was to determine how much air would be entrained by the agent in a standard mortar so that comparisons could be made with other air-entraining agents if so desired.

Investigation No. 3

Air Content Obtained by Using 79-1G Sand in its Natural Gradation.

This investigation was made to determine how much air was entrained by the 79-1G sand, in its natural gradation, for comparison purposes. Three batches were made with the air-entraining agent and two without. Because of the mixer's limitations, no particles were used which were retained on the Number 8 sieve. From the sieve analysis of the 79-1G sand, as shown in Table 1, the gradation of that passing the No. 8 sieve and excluding that passing the No. 200 sieve was determined to be as follows:

<u>Passing Sieve</u>	<u>Retained on Sieve</u>	<u>Percent</u>
No. 8	No. 16	28
No. 16	No. 30	32
No. 30	No. 50	30
No. 50	No. 100	8
No. 100	No. 200	2

* The concentration of air-entraining agent used is explained in the section titled Mix Proportions.

The reason for excluding the material passing the No. 200 sieve is that this material has such a high specific surface that even small changes in the amount of this material could radically affect the results of the mixes.

Investigation No. 4

Mixes to Determine if Fineness Modulus and Specific Surface are Sufficient Gradation Controls to Guarantee a Specified Air Content. This investigation was conducted to determine if different gradations with the same fineness modulus and specific surface would yield the same air content. Nine gradations were chosen as shown in Table 5.

TABLE 5
AGGREGATE GRADATIONS FOR INVESTIGATION NO. 4

Aggregate Gradation Number	Fineness Modulus	Specific Surface ($\text{cm.}^2/\text{gm.}$)	Percent Retained on Sieve Number					
			8	16	30	50	100	200
1	2.40	60	0	2	63	77	98	100
2	2.40	60	0	20	40	80	100	100
3	2.40	60	0	28	30	82	100	100
4	2.70	55	0	35	62	73	100	100
5	2.70	55	0	30	70	70	100	100
6	2.70	55	0	45	47	78	100	100
7	3.00	50	0	60	70	70	100	100
8	3.00	50	0	38	85	85	92	100
9	3.00	50	0	30	90	90	90	100

Three combinations of fineness modulus and specific surface were used:

1. F.M. = 2.40, S.S. = 60 cm.²/gm.
2. F.M. = 2.70, S.S. = 55 cm.²/gm.
3. F.M. = 3.00, S.S. = 50 cm.²/gm.

Three batches of mortar were mixed for each aggregate gradation and the air contents determined. The order of mixing was determined randomly as explained in Appendix B.

Investigation No. 5

Mixes Made With 79-1G Sand, Varying the Fineness Modulus and Specific Surface; With Air-entraining Agent. The purpose of this investigation was to attempt to find a relationship or trend between air content and average particle size as measured by both fineness modulus and specific surface. Twelve aggregate gradations were selected for this study, varying fineness modulus and specific surface in the following manner:

1. F.M. = 2.40, S.S. = 50, 55, 60 and 65 cm.²/gm.
2. F.M. = 2.70, S.S. = 45, 50, 55 and 60 cm.²/gm.
3. F.M. = 3.00, S.S. = 40, 45, 50 and 55 cm.²/gm.

These gradations are shown in Table 6. Three batches were mixed for each gradation. The order of mixing was determined randomly as explained in Appendix B.

TABLE 6

AGGREGATE GRADATIONS FOR INVESTIGATIONS NO. 5, NO. 6 and NO. 7

Aggregate Gradation Number	Fineness Modulus	Specific Surface ($\text{cm.}^2/\text{gm.}$)	Percent Retained on Sieve Number					
			<u>8</u>	<u>16</u>	<u>30</u>	<u>50</u>	<u>100</u>	<u>200</u>
1	2.40	50	0	0	40	100	100	100
2	2.40	55	0	10	40	90	100	100
3	2.40	60	0	2	63	77	98	100
4	2.40	65	0	19	60	61	100	100
5	2.70	45	0	5	75	90	100	100
6	2.70	50	0	31	50	90	99	100
7	2.70	55	0	35	62	73	100	100
8	2.70	60	0	28	70	80	92	100
9	3.00	40	0	28	85	87	100	100
10	3.00	45	0	35	80	90	95	100
11	3.00	50	0	38	85	85	92	100
12	3.00	55	0	48	80	82	90	100

Investigation No. 6

Mixes Made With 79-1G Sand, Varying the Fineness Modulus and Specific Surface; Without Air-entraining Agent. In this investigation the same aggregate gradations were used as in Investigation No. 5, but no air-entraining agent was added to the mixing water. There were two reasons for this investigation. The first was to see if the "entrapped" air contents would follow the same trends as the "total" air contents obtained in Investigation No. 5, and the second was to see if any relationship is displayed between "net" entrained air content and the two measures of average particle size.

Two batches were mixed for each of the gradations listed in Table 6, and the order of mixing was determined randomly.

Investigation No. 7

Mixes Made With Quartzite Sand, Varying the Fineness Modulus and Specific Surface; With Air-entraining Agent. The gradations used in this investigation were the same as those of Investigation No. 5. However, a quartzite sand was used instead of the 79-1G sand. The purpose of this investigation was to determine if the two sands would yield similar or identical results. If so, then it could be reasonably assumed that the results were due primarily to the gradation conditions rather than some other factor, such as mineralogical composition of the sand.

In this investigation three batches were made for each gradation and the order of mixing was determined randomly.

It should be stated that the values of specific surface for the quartzite sand will not be the same as those found for the 79-1G sand. However, since the same gradations were used in this investigation as were used in Investigation No. 5, it can be reasonably assumed that there would be a good relative relationship between the specific surfaces of the two types of sand.

Investigation No. 8

Mixes of One-sized Gradations. The purpose here was to determine how much air would be entrained in batches of mortar made with one-sized aggregate gradations. The following single size fractions were investigated:

<u>Passing Sieve</u>	<u>Retained on Sieve</u>
No. 8	No. 16
No. 16	No. 30
No. 30	No. 50
No. 50	No. 100

Three batches were mixed for each size, with the order of mixing determined randomly.

Investigation No. 9

Mixes Made With 79-1G Sand, Varying the Fineness Modulus and the Percent of Various Size Fractions. The purpose of this investigation was twofold: it was intended to first determine if there was a correlation between the percent of particular size fractions in the gradation and the resulting air content, and then to determine if fineness modulus had any effect on the correlations. To accomplish these purposes it was decided to vary the percent of four size fractions in increments of 10 percent at each of three fineness moduli. The size fractions were No. 8 - No. 16, No. 16 - No. 30, No. 30 - No. 50 and No. 50 - No. 100, and the fineness moduli were 2.40, 2.70 and 3.00. For each combination of fineness modulus and size fraction the following amounts of the size fraction were included in a batch: 0 percent, 10 percent, 20 percent, 30 percent, 40 percent and 50 percent, except that gradations could not be set up for the following combinations:

1. fineness modulus = 2.70, 50 percent of No. 50 - No. 100
2. fineness modulus = 3.00, 40 percent of No. 50 - No. 100
3. fineness modulus = 3.00, 50 percent of No. 50 - No. 100

The gradations listed in Table 7 were used for this investigation. Two batches were mixed for each gradation and, as before, the order of mixing was determined randomly.

Whenever a particular combination of fineness modulus and percent of a certain size fraction was present in more than one gradation, the gradation chosen for the data was determined by random selection.

TABLE 7

AGGREGATE GRADATIONS FOR INVESTIGATION NO. 9

Aggregate Gradation Number	Fineness Modulus	Percent Retained on Sieve Number					
		8	16	30	50	100	200
1	2.40	0	0	50	90	100	100
2	2.40	0	10	40	90	100	100
3	2.40	0	20	40	80	100	100
4	2.40	0	30	40	70	100	100
5	2.40	0	40	50	50	100	100
6	2.40	0	50	50	60	80	100
7	2.40	0	40	40	60	100	100
8	2.40	0	0	40	100	100	100
9	2.70	0	0	70	100	100	100
10	2.70	0	10	70	90	100	100
11	2.70	0	20	70	80	100	100
12	2.70	0	30	70	70	100	100
13	2.70	0	40	40	90	100	100
14	2.70	0	50	60	60	100	100
15	2.70	0	30	50	90	100	100
16	2.70	0	20	50	100	100	100
17	3.00	0	0	100	100	100	100
18	3.00	0	10	90	100	100	100
19	3.00	0	20	80	100	100	100
20	3.00	0	30	80	90	100	100
21	3.00	0	40	70	90	100	100
22	3.00	0	50	60	90	100	100
23	3.00	0	50	50	100	100	100
24	3.00	0	40	60	100	100	100
25	3.00	0	40	80	80	100	100
26	3.00	0	60	70	70	100	100

Mix Proportions

The mix proportions selected were those given in ASTM C 185-59, Standard Method of Test for Air Content of Hydraulic Cement Mortar. The aggregate-cement ratio was 4 to 1, consisting of 1400 gms. of sand and 350 gms. of cement. Enough water was added to the batches to give a flow value between 80 and 95 percent, thus using consistency as a criterion for similar batches. The concentration of air-entraining agent was set at 1 oz. of agent per sack of cement. A fresh solution was made each day, containing 10 ml. of the agent and 425 ml. of dimineralized water. Then 10 ml. of this solution was added to each batch as part of the mixing water. The aggregate gradations desired were obtained by combining, by weight, the proper amounts of the various size fractions.

The above proportions were used for all batches except in Investigation No. 6, where no air-entraining agent was used.

Mixing Procedure

In order to maintain some degree of control of ambient conditions when mixing, all of the mixing was done in an air-conditioned laboratory room. The temperature of this room was maintained at $24 \pm 4^{\circ}\text{C}$ ($75 \pm 7^{\circ}\text{F}$). The materials to be mixed were always stored in this room overnight prior to the day of mixing.

The mixing procedure followed fairly closely the procedure described for mixing mortars in ASTM C 305-59T, Tentative Method for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency. Briefly, the procedure was as follows:

- (1) Place all the cement in the bowl.

- (2) Add the mixing water; then start the mixer and mix at the slow speed (140 ± 5 rpm) for 30 sec.
- (3) Add the entire quantity of sand slowly over a 30-sec. period, while mixing at slow speed.
- (4) Stop the mixer, change to medium speed (285 ± 10 rpm), and mix for 30 sec.
- (5) Stop the mixer and let the mortar stand for 1 1/2 minutes. During the first part of this period scrape down into the batch any mortar that may have collected on the side of the bowl.
- (6) Finish by mixing for one minute at medium speed.

Immediately after mixing, a flow determination was made on the batch according to ASTM C 185-59, Standard Method of Test for Air Content of Hydraulic Cement Mortar. If the flow was not within the required limits of 80 to 95 percent, the batch was discarded. If the flow requirements were met, then the air content of the batch was determined as follows:

- (1) Using that portion of the mortar not used for the flow determination, the bowl of the air meter was filled in three equal layers, starting seven minutes after the mixing of the batch had begun. Each layer was "rodded" 15 times with a 1/8" round glass rod with a smooth rounded tip. When rodding the first layer, care was taken not to strike forcibly against the bottom of the bowl. In rodding the second and third layers, only enough force was used to cause the rod to penetrate the surface of the previous layer.

- (2) The top of the air meter was then fitted over the bowl and filled to the zero level with distilled water. One drop of n-octyl alcohol ($\text{CH}_3(\text{CH}_2)_6\text{CH}_2\text{OH}$) was then added as an anti-foaming agent.
- (3) Placing a thumb over the hole at the top of the meter, the meter was then inverted and agitated vigorously until all the mortar was thoroughly dispersed.
- (4) Next the meter was set upright on a laboratory bench to let the air rise out. The air content was read in milliliters fifteen minutes after the mixing of the batch had begun. By the use of a calibration factor for the bowl, this value in milliliters was converted into the direct percentage of air in the mortar.
- (5) At the time of the air content reading the room temperature was also recorded.

Experimental Design

An attempt was made to set up the experimental designs for this research in such a way that standard statistical techniques could be used to analyze the data. Complete randomization was used in determining the order of mixing the various batches for each particular investigation. The reason for this randomization was to minimize the effect of any extraneous factors (human elements or otherwise) which might have affected the data being gathered.

RESULTS

Investigation No. 1

Preliminary Investigation of Mixer Interaction and the Effect of Fineness Modulus and Specific Surface on Air Entrainment. As mentioned in the Procedure, there were two reasons for this investigation. The first was to see if two mixers would give identical air contents in batches of mortar which have the same aggregate gradations and flow values. The second purpose was to determine if fineness modulus and/or specific surface have any effect on air entrainment.

The two mixers used were designated Mixer No. 1 and Mixer No. 2, Mixer No. 1 meeting ASTM specifications and Mixer No. 2, not meeting ASTM specifications.

The data obtained in this investigation are shown in Table 8, and a graphical representation of air content versus specific surface at the three levels of fineness moduli is shown in Figure 3.

The experimental design of this experiment was set up as a three-factor factorial with all factors fixed and a completely randomized design.* The statistical model is then

$$Y_{ijklm} = \mu + M_i + F_j + MF_{ij} + S_k + MS_{ik} + FS_{jk} + MFS_{ijk} + \epsilon_m(ijk)$$

where

Y_{ijklm} represents the m-th observation of treatment ijk .

μ represents the mean effect

* The statistics references used throughout this study and analysis are listed as references 27 and 28 in the bibliography.

- M_i represents mixer type, $i = 1, 2$
 F_j represents fineness modulus, $j = 1, 2, 3$
 MF_{ij} represents the interaction between M and F
 S_k represents the specific surface, $k = 1, \dots, 4$
 MS_{ik} represents the interaction between M and S
 FS_{jk} represents the interaction between F and S
 MFS_{ijk} represents the three-way interaction between M , F and S
 $\epsilon_m(ijk)$ represents the random error within the ijk cell.

Since $m = 1$, the three-way interaction term and error term are confounded. The three-way interaction term is assumed to be zero.

An analysis of variance (ANOVA) was performed on the data, and is presented in Appendix C. The resulting ANOVA table is presented as Table 9. The analysis shows that the effect of mixer type was non-significant, but that there was a mixer type-fineness modulus interaction present at the 1 percent α -level. There was also a mixer type-specific surface interaction present at the 5 percent α -level. Because of the interactions present, it was decided that for all remaining investigations only Mixer No. 1, the mixer meeting ASTM specifications, would be used.

The analysis of variance also showed that fineness modulus was a highly significant factor in determining air content and that the effect specific surface was also significant. The fact that the effect of fineness modulus was highly significant at the 1 percent α -level and the effect of specific surface was significant at the 5 percent α -level indicated that fineness modulus played a larger role in determining air content than does specific surface.

Finally, the fineness modulus-specific surface interaction was found to be highly significant. This means that at different levels of specific surface, the effect of fineness modulus on air content differed and vice versa.

TABLE 8
DATA FOR INVESTIGATION NO. 1

Aggregate Gradation Number*	Fineness Modulus	Specific Surface ($\text{cm.}^2/\text{gm.}$)	Mixer No. 1			Mixer No. 2		
			Water (cc.)	Flow (%)	Air (%)	Water (cc.)	Flow (%)	Air (%)
1	2.40	50	290	85	12.8	290	85	10.8
2	2.40	55	275	89	10.7	270	85	10.5
3	2.40	60	265	88	9.9	265	80	9.7
4	2.40	65	250	85	12.1	250	90	12.1
5	2.70	50	245	91	10.0	245	87	9.1
6	2.70	55	240	83	9.6	240	90	9.4
7	2.70	60	235	85	10.2	230	82	9.2
8	2.70	65	230	82	9.1	230	80	9.2
9	3.00	50	225	88	7.5	230	95	7.7
10	3.00	55	225	88	8.8	225	88	9.9
11	3.00	60	230	84	11.0	230	90	11.8
12	3.00	65	225	83	8.2	235	94	9.2

* Aggregate gradation numbers correspond to those in Table 4.

FIGURE 3. AIR CONTENT VS. SPECIFIC SURFACE
AT DIFFERENT LEVELS OF
FINENESS MODULUS (79 - 1 G SAND)

TABLE 9

ANALYSIS OF VARIANCE TABLE FOR INVESTIGATION NO. 1

<u>Source</u>	<u>Degree of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>Significance</u>
μ	1	2370.094	2370.094	--	
M_i	1	0.070	0.070	0.778	N.S.
F_j	2	15.707	7.854	87.267	**
MF_{ij}	2	2.352	1.176	13.067	**
S_k	3	1.384	0.461	5.122	*
MS_{ik}	3	1.455	0.485	5.389	*
FS_{jk}	6	21.110	3.518	39.089	**
$\epsilon_{12}(ijk)$	6	0.538	0.090	--	
Totals	24	2412.710	--	--	

M_i represents mixer type, $i = 1, 2$ N.S. = non-significant

F_j represents fineness modulus, $j = 1, 2, 3$ * = significant at 5 percent α -level

S_k represents specific surface, $k = 1, \dots, 4$ ** = significant at 1 percent α -level

Combinations of above letters represent the corresponding interactions.

Investigation No. 2

Air Content of Standard Mortar. The purpose of this investigation was to determine how much air would be entrained in a standard mortar (ASTM C 185-59) by the vinsol-resin air-entraining agent used throughout this project. Hence, these results might be compared with results obtained by using a different air-entraining agent.

The results are given in Table 10, and the corresponding calculations are presented in Appendix D. The average air content of the three batches was 14.8 percent.

TABLE 10
DATA FOR INVESTIGATION NO. 2

<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>
1	210	80	15.0
2	220	86	14.5
3	225	87	14.8

Investigation No. 3

Air Content Obtained by Using 79-1G Sand in its Natural Gradation.

In this investigation the 79-1G sand was used in its natural gradation, as shown in Table 1. The results are given in Table 11. The average air content of the batches using the air-entraining agent was 7.9 percent and the average without agent was 1.9 percent. Thus, a net value of 6.0 percent was added by using the agent.

TABLE 11

DATA FOR INVESTIGATION NO. 3

<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>
1*	240	81	7.4
2*	245	81	8.2
3*	245	86	8.0
4**	265	89	1.8
5**	265	88	2.0

* With air-entraining agent.

** Without air-entraining agent.

As will be seen later, many of the gradations used in this report resulted in air contents higher than 7.9 percent. Thus, it can be concluded that the natural gradation of the 79-1G sand is not the gradation which will result in the highest air contents.

Investigation No. 4

Mixes to Determine if Fineness Modulus and Specific Surface are Sufficient Gradation Controls to Guarantee a Specified Air Content. This investigation was set up to determine if different gradations with the same fineness modulus and specific surface would yield the same air content. The gradations listed in Table 5 were used. There were three gradations for each of three different combinations of fineness modulus and specific surface. Three batches were mixed for each of the nine different gradations. The results are shown in Table 12, and represented graphically in Figure 4. A one-way analysis of variance was performed on the data from each of the combinations of fineness modulus and specific surface and is presented in Appendix E. The statistical model is

$$Y_{ij} = \mu + T_i + \epsilon_{ij}$$

where

Y_{ij} represents the j -th observation of the i -th treatment,
 $j = 1, 2, 3$

μ represents the mean effect

T_i represents the gradation, $i = 1, 2, 3; 4, 5, 6; 7, 8, 9$

ϵ_{ij} represents the random error within cell i

The ANOVA tables resulting from the calculations are presented in Table 13. The analysis shows that there can be a significant difference in the air contents from different gradations of the same fineness modulus and specific surface. Therefore, it can be said that fineness modulus and specific surface are not enough gradation control to pre-determine the resulting air content. This statement must be kept in mind when considering the results of Investigations No. 5 and No. 6.

TABLE 12
DATA FOR INVESTIGATION NO. 4

<u>Aggregate Gradation Number*</u>	<u>Fineness Modulus</u>	<u>Specific Surface (cm.²/gm.)</u>	<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
1	2.40	60	1	265	93	10.2	9.83
			2	260	87	9.9	
			3	260	84	9.4	
2	2.40	60	1	265	88	9.9	8.53
			2	265	89	8.0	
			3	265	89	8.0	
3	2.40	60	1	265	88	8.2	7.80
			2	265	83	8.0	
			3	265	81	7.2	
4	2.70	55	1	245	94	7.8	7.87
			2	240	88	8.0	
			3	240	90	7.8	
5	2.70	55	1	240	83	9.6	7.60
			2	245	93	6.7	
			3	240	82	6.5	
6	2.70	55	1	245	88	6.3	6.60
			2	245	88	6.8	
			3	245	88	6.7	
7	3.00	50	1	225	87	7.2	7.00
			2	225	87	6.3	
			3	225	88	7.5	
8	3.00	50	1	230	85	8.8	9.37
			2	230	81	9.4	
			3	235	90	9.9	
9	3.00	50	1	235	80	10.4	11.07
			2	240	88	11.1	
			3	240	88	11.7	

* Aggregate gradation numbers correspond to those of Table 5.

FIGURE 4. COMPARISON OF AIR CONTENTS OF MIXES USING DIFFERENT AGGREGATE GRADATIONS HAVING THE SAME FINENESS MODULUS AND SPECIFIC SURFACE (79-1 G SAND)

* Aggregate Gradation Numbers Correspond to Those in Table 5.

TABLE 13

ANALYSIS OF VARIANCE TABLES FOR INVESTIGATION NO. 4

ANOVA Table - Gradations 1,2,3

<u>Source</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>Significance</u>
μ	1	689.938	689.938	--	
T_i	2	6.269	3.135	5.710	**
ϵ_{ij}	6	3.293	0.549	--	
Totals	9	699.500	--		

ANOVA Table - Gradations 4,5,6

<u>Source</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>Significance</u>
μ	1	486.938	486.938	--	
T_i	2	2.675	1.338	1.298	N.S.
ϵ_{ij}	6	6.187	1.031	--	
Totals	9	495.800	--		

TABLE 13 (Cont'd)

ANOVA Table - Gradations 7, 8, 9

Source	Degrees of Freedom	Sum of Squares	Mean Square	F-ratio	Significance
μ	1	750.588	750.588	--	
T_1	2	26.029	13.015	33.642	**
ϵ_{1j}	6	12.233	2.039	--	
Totals	9	779.850	--		

 T_1 represents gradation, $i = 1, 2, 3; 4, 5, 6; 7, 8, 9$

N.S. = non-significant

** = significant at one percent α -level

Investigation No. 5

Mixes Made With 79-1G Sand, Varying the Fineness Modulus and Specific Surface; With Air-entraining Agent. In this investigation the air contents were determined for three batches of each of the gradations listed in Table 6. The resulting data are presented in Table 14. A multiple linear regression analysis was performed on the data and is presented in Appendix F. The following regression equation resulted:

$$A = 54.36326 - 13.73406F - 0.6975901S + 0.2032206FS$$

where A = air content, percent,

F = fineness modulus, and

S = specific surface, cm^2/gm .

The correlation index, R^2 , for this equation was 0.6085, which means that approximately 61 percent of the variability in the data can be accounted for by the straight lines described by the above equation. Figure 5 shows the empirical data along with the regression lines. This figure shows that a trend does exist; that is, for a particular value of fineness modulus, the amount of air entrained decreases as the specific surface increases.

It must be remembered that Investigation No. 4 shows that fineness modulus and specific surface alone are not enough gradation control to insure a certain air content. Thus, the above equation will hold true only for the gradations used in this investigation. A different set of aggregate gradations, even though the fineness moduli and specific surfaces were the same, would probably result in a different equation.

TABLE 14
DATA FOR INVESTIGATION NO. 5

<u>Aggregate Gradation Number*</u>	<u>Fineness Modulus</u>	<u>Specific Surface (cm.²/gm.)</u>	<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
1	2.40	50	1	290	80	10.7	11.20
			2	295	85	11.3	
			3	300	93	11.6	
2	2.40	55	1	270	85	10.0	9.93
			2	270	83	9.6	
			3	270	88	10.2	
3	2.40	60	1	265	93	10.2	9.83
			2	260	87	9.9	
			3	260	84	9.4	
4	2.40	65	1	250	83	7.1	7.17
			2	255	85	7.2	
			3	255	80	7.2	
5	2.70	45	1	265	91	10.8	10.80
			2	260	86	11.3	
			3	260	81	10.3	
6	2.70	50	1	245	85	9.2	8.87
			2	245	84	8.5	
			3	245	85	8.9	
7	2.70	55	1	245	94	7.8	7.87
			2	240	88	8.0	
			3	240	90	7.8	
8	2.70	60	1	235	80	8.8	8.73
			2	240	87	8.6	
			3	240	95	8.8	
9	3.00	40	1	245	85	9.6	9.20
			2	245	90	9.2	
			3	245	85	8.8	
10	3.00	45	1	230	84	10.2	9.80
			2	230	81	9.6	
			3	235	84	9.6	
11	3.00	50	1	230	85	8.8	9.37
			2	230	81	9.4	
			3	235	90	9.9	
12	3.00	55	1	225	80	7.7	8.37
			2	230	85	8.5	
			3	230	91	8.9	

* Aggregate gradation numbers correspond to those of Table 6.

FIGURE 5. COMPARISON OF EMPIRICAL DATA AND REGRESSION LINES FOR AIR CONTENT AS A FUNCTION OF FINENESS MODULUS AND SPECIFIC SURFACE (79-16 SAND)

Investigation No. 6

Mixes Made With 79-1G Sand, Varying the Fineness Modulus and Specific Surface; Without Air-entraining Agent. In this investigation an attempt was made to measure the so-called "entrapped" air content of batches of mortar using the same gradations as in Investigation No. 5. The resulting data are presented in Table 15, and the curves are shown in Figure 6. It can be seen that these curves do not display the same trends shown in Figure 5.

The average air contents of this investigation were subtracted from the corresponding average air contents of Investigation No. 5 to give values for "net" entrained air due to the air-entraining agent. That is, "total" air content with agent minus "entrapped" air content without agent equals "net" entrained air due to addition of agent.

FIGURE 6. "ENTRAPPED" AIR CONTENT VS.
SPECIFIC SURFACE AT DIFFERENT
LEVELS OF FINENESS MODULUS
(79-1G SAND)

The resulting values are given in Table 16, and the curves are shown as Figure 7. These curves seem to display a fairly good straight-line relationship, following the trend displayed in Figure 5.

A multiple correlation analysis was performed on this latter data and the following equation resulted:

$$A = 48.61519 - 11.46800F - 0.57197095S + 0.1363603FS$$

where A = "net" air content, percent,

F = fineness modulus, and

S = specific surface, cm^2/gm .

The correlation index for this equation is 0.8956. The calculations for this analysis are presented in Appendix G. Figure 7 shows the prediction curves along with the curves from the data. Once again, it must be remembered that these curves could differ for different gradations with the same fineness moduli and specific surfaces. The preciseness of the above curves leads one to believe that the amount of air entrained in a mix does not depend too much on the amount of entrapped air, but rather, is a direct function of the gradation of the aggregate. Perhaps Investigation No. 9 will shed more light on the subject of air content as a function of aggregate gradation characteristics.

TABLE 15
DATA FOR INVESTIGATION NO. 6

<u>Aggregate Gradation Number*</u>	<u>Fineness Modulus</u>	<u>Specific Surface (cm.²/gm.)</u>	<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
1	2.40	50	1	330	92	1.9	2.05
			2	325	87	2.2	
2	2.40	55	1	305	91	2.2	2.35
			2	305	94	2.5	
3	2.40	60	1	285	92	2.7	3.00
			2	285	92	3.3	
4	2.40	65	1	280	92	2.5	2.20
			2	275	91	1.9	
5	2.70	45	1	300	95	1.9	2.05
			2	285	85	2.2	
6	2.70	50	1	275	82	2.5	2.25
			2	270	90	2.0	
7	2.70	55	1	260	87	1.7	2.05
			2	250	85	2.4	
8	2.70	60	1	260	90	3.5	3.00
			2	250	83	2.5	
9	3.00	40	1	275	90	1.6	1.80
			2	265	90	2.0	
10	3.00	45	1	260	85	2.7	2.45
			2	260	82	2.2	
11	3.00	50	1	265	95	3.1	3.05
			2	260	93	3.0	
12	3.00	55	1	250	95	3.1	3.10
			2	245	88	3.1	

* Aggregate gradation numbers correspond to those of Table 6.

TABLE 16
 "NET" ENTRAINED AIR CONTENTS

<u>Aggregate Gradation Number*</u>	<u>Average Air Content With Agent</u>	<u>Average Air Content Without Agent</u>	<u>"Net" Air Content</u>
1	11.20	2.05	9.15
2	9.93	2.35	7.58
3	9.83	3.00	6.83
4	7.17	2.20	4.97
5	10.80	2.05	8.75
6	8.87	2.25	6.62
7	7.87	2.05	5.82
8	8.73	3.00	5.73
9	9.20	1.80	7.40
10	9.80	2.45	7.35
11	9.37	3.05	6.32
12	8.37	3.10	5.27

* Aggregate gradation numbers correspond to those of Table 6.

FIGURE 7. COMPARISON OF EMPIRICAL DATA AND REGRESSION LINES FOR "NET" ENTRAINED AIR CONTENT AS A FUNCTION OF FINENESS MODULUS AND SPECIFIC SURFACE (79-1G SAND)

Investigation No. 7

Mixes Made With Quartzite Sand, Varying the Fineness Modulus and Specific Surface; with Air-entraining Agent. The purpose of this investigation was to see if two different sands would give similar results in mixes using the same gradations.

The data obtained in this investigation are presented in Table 17. A multiple linear regression analysis was performed on the data and is presented in Appendix H. The analysis yielded the following regression equation with a correlation index, R^2 of 0.7570:

$$A = 16.50663 + 2.096219F - 0.01579947S - 0.08682261FS$$

where A = air content, percent,

F = fineness modulus, and

S = specific surface, cm^2/gm .

The empirical data and regression lines are presented in Figure 8.

Figure 9 gives a comparison of the empirical data obtained with the two sands. Although there appears to be some similarity between the two sets of curves, the above regression equation appears to be quite different from the equation of Investigation No. 5. Figure 10 shows the regression curves from both investigations.

A "students" t-test on the difference of means was performed on the data for each of the three fineness moduli. The results are presented in Appendix H. The following interpretations were made:

- a. At the fineness modulus of 2.40, there was no significant difference of means at the one percent α -level, but there was a significant difference at the five percent α -level.

- b. At the fineness modulus of 2.70, there was no significant difference of means at the 5 percent α -level.
- c. At the fineness modulus of 3.00, there was no significant difference of means at the 5 percent α -level.

Next, a t-test was performed on the difference of slopes of the prediction equations from the two investigations at each of the three fineness moduli. These results are also presented in Appendix H.

The following interpretations were made:

- a. At the fineness modulus of 2.40, there is no significant difference in the slopes at the 5 percent α -level.
- b. At the fineness modulus of 2.70, there is no significant difference in the slopes at the 5 percent α -level.
- c. At the fineness modulus of 3.00, there was a significant difference in the slopes at the 1 percent α -level.

Thus, it can be seen that no general statement can be made as to whether or not the two types of sand give the same results. One could not definitely conclude that the results are the same, as there were a few significant differences. On the other hand, there were also quite a few similarities.

TABLE 17
DATA FOR INVESTIGATION NO. 7

<u>Aggregate Gradation Number*</u>	<u>Fineness Modulus</u>	<u>Specific Surface** (cm.²/gm.)</u>	<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
1	2.40	50	1	330	91	10.2	10.60
			2	330	85	11.0	
			3	330	82	10.6	
2	2.40	55	1	310	88	10.4	9.60
			2	310	88	9.0	
			3	310	90	9.4	
3	2.40	60	1	275	87	8.2	8.93
			2	275	89	8.4	
			3	275	93	10.2	
4	2.40	65	1	280	95	6.8	6.47
			2	275	88	6.5	
			3	275	90	6.1	
5	2.70	45	1	270	91	11.3	11.07
			2	270	94	10.2	
			3	275	95	11.7	
6	2.70	50	1	265	94	8.6	8.67
			2	275	85	9.2	
			3	275	80	8.2	
7	2.70	55	1	255	84	6.8	7.03
			2	255	82	6.7	
			3	260	95	7.6	
8	2.70	60	1	255	80	6.8	7.33
			2	260	92	7.6	
			3	255	93	7.6	
9	3.00	40	1	260	91	11.5	11.83
			2	255	93	11.9	
			3	255	92	12.1	
10	3.00	45	1	240	81	9.8	9.93
			2	245	85	9.2	
			3	245	94	10.8	
11	3.00	50	1	250	90	10.0	10.00
			2	250	92	10.4	
			3	250	93	9.6	
12	3.00	55	1	245	87	7.8	7.93
			2	245	89	8.0	
			3	245	90	8.0	

* Aggregate gradation numbers correspond to those of Table 6.

** Relative values only.

FIGURE 8. COMPARISON OF EMPIRICAL DATA AND REGRESSION LINES FOR AIR CONTENT AS A FUNCTION OF FINENESS MODULUS AND SPECIFIC SURFACE (QUARTZITE SAND)

FIGURE 9. COMPARISON OF RESULTS OBTAINED WITH IDENTICAL GRADATIONS OF 79-1G SAND AND QUARTZITE SAND

FIGURE 10. COMPARISON OF REGRESSION LINES FOR 79-1G SAND AND QUARTZITE SAND

Investigation No. 8

Mixes of One-sized Gradations. The purpose of this investigation was to determine if different single-sized fractions of sand entrain different amounts of air, and if so, which sizes differ. The results obtained are given in Table 18, and are shown in Figure 11.

TABLE 18

DATA FOR INVESTIGATION NO. 8

<u>Passing Sieve Number</u>	<u>Retained on Sieve Number</u>	<u>Batch Number</u>	<u>Water (c.c.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
8	16	1	250	86	9.4	9.5
		2	250	80	7.8	
		3	255	88	11.3	
16	30	1	270	90	12.4	11.9
		2	285	90	10.3	
		3	285	90	13.0	
30	50	1	310	85	12.4	12.7
		2	310	82	14.9	
		3	315	80	10.7	
50	100	1	400	87	7.1	8.0
		2	400	86	8.0	
		3	400	85	8.9	

A one-way analysis of variance was performed on the data, using the following statistical model:

$$Y_{ij} = \mu + T_i + \epsilon_{ij}$$

where

Y_{ij} represents the j -th observation of the i -th treatment,

$j = 1, 2, 3$

μ represents the mean effect

T_i represents the size fraction, $i = 1, 2, 3, 4$

ϵ_{ij} represents the random error within cell i

FIGURE II. COMPARISON OF AIR CONTENTS
OBTAINED WITH SINGLE SIZE
FRACTIONS OF 79-1 G SAND

The ANOVA table is given in Table 19, and shows that there is a significant difference in the amount of air entrained by the separate size-fractions.

TABLE 19

ANALYSIS OF VARIANCE TABLE FOR INVESTIGATION NO. 8

<u>Source</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>Significance</u>
μ	1	1327.203	1327.203	--	
T_i	2	41.710	20.063	8.966	**
ϵ_{ij}	9	20.707	2.301	--	
Totals	12	1389.620	--		

T_i represents size fraction, $i=1, \dots, 4$

** = significant at one percent α -level.

A test on means (Duncan test) showed which sizes were significantly different and the levels of significance. These were:

1. Sizes No. 30-50 and No. 50-100 were significantly different at the one percent level.
2. Sizes No. 8-16 and No. 30-50 were significantly different at the five percent level.
3. Sizes No. 16-30 and No. 50-100 were significantly different at the five percent level.

There is no significant difference in the amount of air entrained by the following pairs of sizes:

1. Sizes No. 8-16 and No. 16-30.
2. Sizes No. 16-30 and No. 30-50.
3. Sizes No. 8-16 and No. 50-100.

The statistical analysis for this investigation is presented in

Investigation No. 9

Mixes Made With 79-1G Sand, Varying the Fineness Modulus and the Percent of Various Size Fractions. The purpose of this investigation was to determine what effect, if any, the percent of sand of certain size fractions would have on air content. The size fractions investigated were No. 8 - No. 16, No. 16 - No. 30, No. 30 - No. 50 and No. 50 - No. 100.

The data obtained is presented in Table 20, and Figures 12, 13, 14 and 15 show the resulting curves of air content versus the percent of sand of a particular size fraction, for all three fineness moduli.

In order to set up a factorial arrangement for a three-way analysis of variance, the 40 and 50 percent values of percent of size fraction present were dropped.

The statistical model used here was:

$$Y_{ijklm} = \mu + A_i + B_j + AB_{ij} + C_k + AC_{ik} + BC_{jk} + ABC_{ijk} + \epsilon_m(ijk)$$

where

Y_{ijklm}	represents the m-th observation of treatment ijk , $m = 1, 2$
μ	represents the mean effect
A_i	represents size fraction, $i = 1, 2, 3, 4$
B_j	represents fineness modulus, $j = 1, 2, 3$
AB_{ij}	represents the interaction between A and B
C_k	represents the percent of the size fraction present in the aggregate gradation, $k = 1, 2, 3, 4$
AC_{ik}	represents the interaction between A and C
BC_{jk}	represents the interaction between B and C
ABC_{ijk}	represents the three-way interaction between A, B and C
$\epsilon_m(ijk)$	represents the random error within the ijk cell

The analyses for this investigation are presented in Appendix J. Table 21 contains the results of the three-way ANOVA. The results show that all factors (size fraction, fineness modulus and percent of size fraction present) and all interactions were significant at the one-percent α -level. Since the first factor, size fraction, was found to be highly significant, and also to include as much of the data as possible, the data was analyzed separately for each size fraction. In this case a two-way analysis of variance was used.

The percent of size fraction present for the first three size fractions ranged from 0 to 50 percent, whereas, for the size fraction No. 50 - No. 100, the range was from 0 to 30 percent. Table 22 presents the ANOVA tables from this analysis. In this case Factor A_i represents the fineness modulus and B_j represents the percent of the size-fraction present in the aggregate gradation. As can be seen in Table 22, for all size fractions the factor representing percent of size fraction present in the aggregate gradation was found to be significant at the one percent α -level. All of the main effects were tested for linear and quadratic significance (presented in Appendix J). In all cases except one, the linear effects were significant at the one percent α -level; and the excepted case was significant at the ten percent α -level. On the contrary, in four cases the quadratic effect was non-significant at the ten percent α -level, and in only two cases was it significant at the one percent α -level.

Due to the fact that almost all linear effects were significant at the one percent α -level, it was decided to perform a multiple linear regression analysis on the data. The results of this analysis are presented in the Appendix, and Figures 16, 17, 18 and 19 give a comparison

between the empirical data and the regression lines. As can be seen, the best relationship was found between air content and the percent of sand in the No. 8 - No. 16 size range. The next best was with the No. 16 - No. 30 size range, then No. 50 - No. 100. The poorest relationship was between air content and the No. 30 to No. 50 size range, in which case only 36 percent of the variation in the data could be accounted for by the prediction equation.

The following are the prediction equations obtained and their correlation index, R^2 , where

A = air content, percent

F = fineness modulus

P = percent of size fraction present in the
aggregate gradation

Size Fraction No. 8 - No. 16:

$$A = 11.73174 - 0.4072302P + 0.1130306FP$$

$$R^2 = 0.83442765$$

Size Fraction No. 16 - No. 30:

$$A = 1.110363 + 2.412789F + 0.4060050P - 0.1276209FP$$

$$R^2 = 0.58085919$$

Size Fraction No. 30 - No. 50

$$A = -9.142344 + 6.230262F + 0.5232462P - 0.1747649FP$$

$$R^2 = 0.35765482$$

Size Fraction No. 50 - No. 100

$$A = 17.53657 - 2.416631F - 0.1030000P$$

$$R^2 = 0.51855893$$

TABLE 20

DATA FOR INVESTIGATION NO. 9

PART I - SIZE FRACTION NO. 8 - NO. 16

<u>Fineness Modulus</u>	<u>Percent of Size Fraction</u>	<u>Aggregate Gradation Number*</u>	<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
2.40	0	1	1	275	85	11.7	12.30
			2	275	91	12.9	
2.40	10	2	1	270	83	9.6	9.90
			2	270	88	10.2	
2.40	20	3	1	265	88	9.9	9.75
			2	265	86	9.6	
2.40	30	4	1	260	82	7.6	7.50
			2	265	92	7.4	
2.40	40	5	1	260	84	6.5	6.50
			2	260	82	6.5	
2.40	50	6	1	255	94	4.5	4.50
			2	250	85	4.5	
2.70	0	9	1	265	81	11.1	10.95
			2	270	90	10.8	
2.70	10	10	1	260	93	11.3	10.85
			2	255	94	10.4	
2.70	20	11	1	245	91	10.0	9.40
			2	245	85	8.8	
2.70	30	12	1	240	83	9.6	8.70
			2	245	95	7.8	
2.70	40	13	1	245	80	8.8	8.80
			2	250	88	8.8	
2.70	50	14	1	245	87	6.3	6.10
			2	245	88	5.9	
3.00	0	17	1	270	90	12.4	11.35
			2	285	90	10.3	
3.00	10	18	1	265	92	10.6	10.30
			2	260	80	10.0	
3.00	20	19	1	255	92	12.9	11.55
			2	250	80	10.2	
3.00	30	20	1	250	93	11.0	10.10
			2	250	91	9.2	
3.00	40	21	1	240	88	9.4	9.50
			2	240	89	9.6	
3.00	50	22	1	235	90	7.6	7.20
			2	235	90	6.8	

TABLE 20 (Cont'd)

PART II - SIZE FRACTION NO. 16 - NO. 30

<u>Fineness Modulus</u>	<u>Percent of Size Fraction</u>	<u>Aggregate Gradation Number*</u>	<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
2.40	0	7	1	260	81	6.8	6.80
			2	265	86	6.8	
2.40	10	4	1	260	82	7.6	7.50
			2	265	92	7.4	
2.40	20	3	1	265	88	9.9	9.75
			2	265	86	9.6	
2.40	30	2	1	270	83	9.6	9.90
			2	270	88	10.2	
2.40	40	8	1	295	85	11.3	11.45
			2	300	93	11.6	
2.40	50	1	1	275	85	11.7	12.30
			2	275	91	12.9	
2.70	0	13	1	245	80	8.8	8.80
			2	250	88	8.8	
2.70	10	14	1	245	87	6.3	6.10
			2	245	88	5.9	
2.70	20	15	1	255	91	8.8	8.90
			2	255	92	9.0	
2.70	30	16	1	260	80	10.8	10.40
			2	265	87	10.0	
2.70	40	12	1	240	83	9.6	8.70
			2	245	95	7.8	
2.70	50	11	1	245	91	10.0	9.40
			2	245	85	8.8	
3.00	0	23	1	240	87	9.2	9.10
			2	240	82	9.0	
3.00	10	22	1	235	90	7.6	7.20
			2	235	90	6.8	
3.00	20	24	1	240	85	9.6	9.30
			2	240	83	9.0	
3.00	30	21	1	240	88	9.4	9.50
			2	240	89	9.6	
3.00	40	25	1	245	93	10.2	9.70
			2	240	90	9.2	
3.00	50	20	1	250	93	11.0	10.10
			2	250	91	9.2	

TABLE 20 (Cont'd)

PART III - SIZE FRACTION NO. 30 - NO. 50

<u>Fineness Modulus</u>	<u>Percent of Size Fraction</u>	<u>Aggregate Gradation Number*</u>	<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
2.40	0	5	1	260	84	6.5	6.50
			2	260	82	6.5	
2.40	10	6	1	255	94	4.5	4.50
			2	250	85	4.5	
2.40	20	7	1	260	81	6.8	6.80
			2	265	86	6.8	
2.40	30	4	1	260	82	7.6	7.50
			2	265	92	7.4	
2.40	40	1	1	275	85	11.7	12.30
			2	275	91	12.9	
2.40	50	2	1	270	83	9.6	9.90
			2	270	88	10.2	
2.70	0	12	1	240	83	9.6	8.70
			2	245	95	7.8	
2.70	10	11	1	245	91	10.0	9.40
			2	245	85	8.8	
2.70	20	10	1	260	93	11.3	10.85
			2	255	94	10.4	
2.70	30	9	1	265	81	11.1	10.95
			2	270	90	10.8	
2.70	40	15	1	255	91	8.8	8.90
			2	255	92	9.0	
2.70	50	16	1	260	80	10.8	10.40
			2	265	87	10.0	
3.00	0	26	1	225	87	7.2	6.75
			2	225	87	6.3	
3.00	10	18	1	265	92	10.6	10.30
			2	260	80	10.0	
3.00	20	19	1	255	92	12.9	11.55
			2	250	80	10.2	
3.00	30	22	1	235	90	7.6	7.20
			2	235	90	6.8	
3.00	40	24	1	240	85	9.6	9.30
			2	240	83	9.0	
3.00	50	23	1	240	87	9.2	9.10
			2	240	82	9.0	

TABLE 20 (Cont'd)

PART IV - SIZE FRACTION NO. 50 - NO. 100

<u>Fineness Modulus</u>	<u>Percent of Size Fraction</u>	<u>Aggregate Gradation Number*</u>	<u>Batch Number</u>	<u>Water (cc.)</u>	<u>Flow (%)</u>	<u>Air (%)</u>	<u>Average Air Content</u>
2.40	0	8	1	295	85	11.3	11.45
			2	300	93	11.6	
2.40	10	1	1	275	85	11.7	12.30
			2	275	91	12.9	
2.40	20	3	1	265	88	9.9	9.75
			2	265	86	9.6	
2.40	30	4	1	260	82	7.6	7.50
			2	265	92	7.4	
2.40	40	7	1	260	81	6.8	6.80
			2	265	86	6.8	
2.40	50	5	1	260	84	6.5	6.50
			2	260	82	6.5	
2.70	0	16	1	260	80	10.8	10.40
			2	265	87	10.0	
2.70	10	15	1	255	91	8.8	8.90
			2	255	92	9.0	
2.70	20	11	1	245	91	10.0	9.40
			2	245	85	8.8	
2.70	30	12	1	240	83	9.6	8.70
			2	245	95	7.8	
2.70	40	14	1	245	87	6.3	6.10
			2	245	88	5.9	
3.00	0	19	1	255	92	12.9	11.55
			2	250	80	10.2	
3.00	10	22	1	235	90	7.6	7.20
			2	235	90	6.8	
3.00	20	25	1	245	93	10.2	9.70
			2	240	90	9.2	
3.00	30	26	1	225	87	7.2	6.75
			2	225	87	6.3	

* Aggregate gradation numbers correspond to those of Table 7.

FIGURE 12. AIR CONTENT VS. THE PERCENT OF SIZE FRACTION
NO. 8 - NO. 16 PRESENT IN GRADATION (79 - 16 SAND)

FIGURE 13. AIR CONTENT VS. THE PERCENT OF SIZE FRACTION NO. 16 - NO. 30 PRESENT IN GRADATION (79 - 16 SAND)

FIGURE 14. AIR CONTENT VS. THE PERCENT OF SIZE FRACTION NO. 30 - NO. 50 PRESENT IN GRADATION (79 - 16 SAND)

FIGURE 15. AIR CONTENT VS. THE PERCENT OF SIZE FRACTION NO. 50 - NO. 100 PRESENT IN GRADATION (79 - 1G SAND)

TABLE 21

THREE-WAY ANOVA TABLE FOR INVESTIGATION NO. 9

<u>Source</u>	<u>Degrees of Freedom</u>	<u>Sums of Squares</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>Significance</u>
A_i	3	49.91375	16.63792	28.17026	***
B_j	2	9.79083	4.89542	8.28861	***
C_k	3	20.89042	6.96347	11.79010	***
AB_{ij}	6	60.28250	10.04708	17.01107	***
AC_{ik}	9	88.56208	9.84023	16.66085	***
BC_{jk}	6	12.22333	2.03722	3.44929	***
ABC_{ijk}	18	63.65657	3.53648	5.98774	***
$\epsilon_m(ijk)$	48	28.35000	0.59062	--	
Totals	95	333.66946	--		

A_i represents size fraction, $i = 1, \dots, 4$

B_j represents fineness modulus, $j = 1, 2, 3$

C_k represents the percent of the size fraction present

in the aggregate gradation, $k = 1, \dots, 4$

Combinations of the above letters represent the corresponding interactions.

*** = significant at one percent α -level.

TABLE 22

TWO-WAY ANOVA TABLES FOR INVESTIGATION NO. 9

Size Fraction No. 8 - No. 16

<u>Source</u>	<u>Degrees of Freedom</u>	<u>Sums of Squares</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>Significance</u>
A_i	2	15.24056	7.62028	11.600	***
B_j	5	117.37472	23.47494	35.734	***
AB_{ij}	10	16.91610	1.69161	2.575	**
$\epsilon_{k(ij)}$	18	11.82500	0.65694	--	
Totals	35	161.35637	--		

Size Fraction No. 16 - No. 30

<u>Source</u>	<u>Degrees of Freedom</u>	<u>Sums of Squares</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>Significance</u>
A_i	2	4.86222	2.43111	6.806	***
B_j	5	54.82222	10.96444	30.694	***
AB_{ij}	10	22.01110	2.20111	6.162	***
$\epsilon_{k(ij)}$	18	6.43000	0.35722	--	
Totals	35	88.12554			

TABLE 22 (Cont'd)

Size Fraction No. 30 - No. 50

Source	Degrees of Freedom	Sums of Squares	Mean Squares	F-ratio	Significance
A_i	2	22.97555	11.48778	23.498	***
B_j	5	38.54222	7.70844	15.767	***
AB_{ij}	10	81.00776	8.10078	16.570	***
$\epsilon_{k(ij)}$	18	8.80000	0.48889	--	
Totals	35	151.32554			

Size Fraction No. 50 - No. 100

Source	Degrees of Freedom	Sums of Squares	Mean Squares	F-ratio	Significance
A_i	2	8.57333	4.28667	6.138	**
B_j	3	26.60333	12.20111	17.472	***
AB_{ij}	6	24.03666	4.00611	5.737	***
$\epsilon_{k(ij)}$	12	8.38000	0.69833	--	
Totals	23	77.59333			

A_i represents fineness modulus, $i = 1, 2, 3$ ** = significant at 5 percent α -level.

B_j represents the percent of the size fraction present in the aggregate gradation, $j = 1, \dots, 4$ *** = significant at 1 percent α -level.

AB_{ij} represents the interaction between the above two factors

FIGURE 16. COMPARISON OF EMPIRICAL DATA AND REGRESSION LINES FOR AIR CONTENT AS A FUNCTION OF FINENESS MODULUS AND THE PERCENT OF SIZE FRACTION NO.8 - NO.16 PRESENT IN GRADATION (79-1G SAND)

FIGURE 17. COMPARISON OF EMPIRICAL DATA AND REGRESSION LINES FOR AIR CONTENT AS A FUNCTION OF FINENESS MODULUS AND THE PERCENT OF SIZE FRACTION NO.16 - NO.30 PRESENT IN GRADATION (79-1G SAND)

FIGURE 18. COMPARISON OF EMPIRICAL DATA AND REGRESSION LINES FOR AIR CONTENT AS A FUNCTION OF FINENESS MODULUS AND THE PERCENT OF SIZE FRACTION NO.30 -NO.50 PRESENT IN GRADATION (79-1G SAND)

FIGURE 19. COMPARISON OF EMPIRICAL DATA AND REGRESSION LINES FOR AIR CONTENT AS A FUNCTION OF FINENESS MODULUS AND THE PERCENT OF SIZE FRACTION NO.50 - NO.100 PRESENT IN GRADATION (79 - 1G SAND)

DISCUSSION OF RESULTS

The results have shown that different mixer paddles (thus different types of mixing action) caused different amounts of air to be entrained in mortars made with identical aggregate gradations. This is not surprising, but it is important for it indicates that the type of mixer should be taken into consideration when designing air entrained concrete mixes. Perhaps if air entrainment records were kept on mixers, they could be used for future mix designs on jobs employing those mixers.

This study supported the fact that variations in fine aggregate gradation do affect air entrainment as stated by Powers (15), Craven (6) and others (8, 13, 5). In particular, air content was found to vary with changes in fineness modulus and specific surface. It was shown that as fineness modulus increased (sand became coarser) the air content decreased. This same relationship was found by Craven (6) and Walker and Bloem (13). As specific surface increased (sand became finer), it was found that the air content decreased at different levels of fineness modulus. These seem to be conflicting results, and lead to the conclusion that the coarseness or fineness of the sand alone cannot be used to predict air content. In fact, it was shown that fineness modulus and specific surface are not enough gradation control to accurately determine air content. A study of interstitial aggregate void characteristics and air entrainment might shed more light on this subject since these void characteristics probably influence the air bubble size and distribution.

It is not suggested that fineness modulus and specific surface are not useful in considerations of air entrainment. These factors are useful measurements of aggregate gradation characteristics and can be used to explain differences in air contents. To explain the causal relationships a study of the physical properties of the materials, not their measurement, is necessary.

This study showed that the total amount of entrained air in mortars is not directly related to the amount of entrapped air. This agrees with the findings of Neville (4), but does not agree with Singh's statement that "the gross entrained air is related to the air entrained under natural conditions; that is, an aggregate that will entrain a large amount of air under natural conditions will entrain proportionally more air with an air-entraining admixture."

The "net" entrained air content (total entrained air minus entrapped air) displayed a good linear correlation with fineness modulus and specific surface, as one was held constant and the other varied. The amount of "net" entrained air was more accurately predicted by fineness modulus and specific surface than was total air content.

The investigation comparing the results of two different sands (natural sand and crushed quartzite sand) gave results which were very similar but not statistically identical. It is felt that the similarity of the results suggests that the variations in air content observed can be attributed in large part to differences in aggregate gradations rather than other characteristics of the two sands.

When taken separately, different size fractions of sand entrain different amounts of air. The fractions No. 16 - No. 30 and No. 30 - No. 50 entrain similar amounts and this amount is more than that entrained by the

fractions No. 8 - No. 16 and No. 50 - No. 100. This is in agreement with statements by the Portland Cement Association (12), Troxell and Davis (14) and Scripture, Hornibrook and Bryant (8).

This study showed that the amount of air entrained in mortar is well correlated with the amount of the size fraction No. 8 - No. 16 present in the aggregate gradation. As the amount of this size fraction increased, the air content decreased. This correlation was better than that for any other size fraction. Air content increased with increases in the amount of size fractions No. 16 - No. 30 and No. 30 - No. 50. This relationship has been reported by Walker and Bloem (13), Craven (6) and Mielenz, Walkodoff, Backstrom and Flack (18). However, the correlation between air content and the amount of No. 30 - No. 50 was very poor. This agrees with results reported by Singh (5). Singh also stated that the size fraction No. 50 - No. 100 is not conducive to air entrainment because "as the amount of material of this size increases there is a marked tendency for the air to decrease" (5). The same relationship was found in this study.

It has often been concluded that the size fraction which entrains the most air when taken separately is the size fraction which has the most influence on air entrainment. This is not necessarily so, since the interstitial void characteristics are a result of all of the size fractions fitted together, and these void characteristics probably have a significant role in the air entrainment process. Powers discussed this idea and stated that "no one size group can have an independent effect in a mixture comprising many sizes" (7). Thus, as shown in this investigation, even though certain fractions entrain more air when taken separately, it cannot be assumed that these fractions display the most influence on the amount of air in mortar and concrete.

Since this study dealt only with portland cement mortar, no definite conclusions can be drawn as to how the results obtained will be reflected in concrete mixes. As has been previously stated, however, the sand fraction of a concrete aggregate is that part of the aggregate which has the most influence on air entrainment in concrete. Therefore, it seems likely that the variations obtained in the air content of mortar might also be presented in concrete. It must be remembered, however, that mortar comprises approximately one-half of the values in concrete. Therefore, a variation in the air content of mortar would be of about one-half the magnitude, percentage-wise, in concrete. Hence, a variation of 3 percent air in mortar caused by sand gradation differences might result in a difference of 1 1/2 percent air in concrete. However, a change of 1 1/2 percent air in concrete might be all that is necessary, when the problem of concern is with marginal air contents.

From the results reported here and in the literature reviewed, it seems appropriate to make the following recommendations:

1. If low air contents are encountered, it would be beneficial to attempt to increase the amount of the No. 16-No. 30 and No. 30-No. 50 fractions in the fine aggregate.

2. If high air contents are encountered, additional amounts of No. 8-No. 16 or No. 50-No. 100 fractions will decrease the air content.

The above statements may not always hold true, and it would be advisable to mix a few trial batches of concrete to be sure the results obtained are those desired.

CONCLUSIONS

Based on the results of this study, the following conclusions can be made.

1. The mixing action produced by different types of paddles on similar mixers will cause different amounts of air to be entrained in mortar mixes with the same aggregate gradations.
2. Both fineness modulus and specific surface influence the amount of air entrained in a mortar.
3. Fineness modulus and specific surface are not enough gradation control to accurately predict air content.
4. The amount of air entrapped in mortar does not follow the same trend as the amount of entrained air.
5. There was a relationship between the amount of air entrained in a mortar and the percent of sand of certain size fractions present in the aggregate gradation. The best relationship is present with the No. 8 - No. 16 size fraction. As the amount of sand in this size fraction increases, the amount of air entrained decreases.

PROPOSED RESEARCH

In light of observations made in this study, the following recommendations seem desirable:

1. Development of a small laboratory mixer for research work, capable of mixing mortars containing particles up to $3/8$ " in diameter.
2. Application of principles stated herein to concrete mixes.
3. Investigation of the possibility of changes in aggregate specifications to allow greater flexibility of fine aggregate gradations for purposes of air content control.
4. Investigation of the variation of bubble size and distribution as caused by variations in fine aggregate gradation characteristics.

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APPENDIX

APPENDIX A
DETERMINATION OF SPECIFIC SURFACE OF
79-1G AGGREGATE FRACTIONS

The method of determining the specific surface of the various size groups of the 79-1G sand aggregate is described in this appendix. The values obtained by this procedure are not the actual values of the specific surface, but are to be recognized as only relative values.

In order to obtain the values for specific surface it was necessary to make a few simplifying assumptions. The first assumption made was that the particles were spherical. This assumption was necessary for the derivation of the specific surface equation. The second assumption was that the particles of a particular size fraction were evenly distributed between the sieve they passed and the sieve they were retained on; thus the average particle diameter of a particular fraction was the average of the sieve openings of the two sieves.*

The "apparent" specific gravity was used in determining the specific surface since this value of specific gravity is determined by using the volume and weight of the solid portion of the aggregate plus impermeable voids. It was felt that this would include all surface area which would be in contact with cement paste.

Singh (21) discussed an angularity factor, f , to be applied to the specific surface value calculated, since the aggregate will never consist of exactly spherical particles. The angularity factor was first introduced by Loudon (24), who suggests that $f = 1.1$ for rounded sand, $f = 1.25$ for

* Exception - material passing the No. 200 sieve. It was assumed that this material would be retained on the No. 400 sieve.

sand of medium angularity, and $f = 1.4$ for angular sand. This means that a personal evaluation must be made to decide which of these three categories the sand in question should be assigned to. Since the sand involved here was neither well-rounded nor highly angular, it was decided to apply the medium angularity factor of $f = 1.25$.

Derivation of Specific Surface Equations

Assuming spherical particles, the following formulas apply:

$$\text{Surface area} = \pi d_a^2 = S_a$$

S_a = surface area of a sphere of diameter d_a

d_a = average diameter (cm.) of sieve openings of two
sieves describing the particular sand fraction
in question

$$\text{Volume} = \pi d_a^3 / 6 = V$$

V = volume of sphere of diameter d_a

$$\text{Weight} = V G_s \gamma_w = W$$

W = weight of sphere of diameter d_a and apparent specific
gravity G_s

γ_w = unit weight of water = 1 in cgs system of units

The specific surface of a sphere, SS , is equal to its surface area divided by its weight. Thus

$$SS = \frac{S_a}{W} = \frac{\pi d_a^2}{\pi d_a^3 / 6 G_s \gamma_w}$$

$$SS = \frac{6}{d_a G_s \gamma_w}$$

$$\text{Since } \gamma_w = 1, SS = \frac{6}{d_a G_s}$$

Applying the angularity factor of $f = 1.25$, the adjusted specific surface, S , is

$$S = 1.25 \times \frac{6}{d_a G_s} = \frac{7.5}{d_a G_s}$$

Example Calculation:

Size fraction No. 8 - No. 16

$$d_{\text{larger}} = .238 \text{ cm}$$

$$d_{\text{smaller}} = .119 \text{ cm}$$

$$\therefore d_a = .1785 \text{ cm}$$

$$G_s = 2.71$$

$$\therefore S = \frac{7.5}{.1785 \times 2.71}$$

$$S = 15.50 \text{ cm}^2 / \text{gm}$$

Determination of Apparent Specific Gravity of 79-1G Aggregate Fractions

The following procedure was used to determine the apparent specific gravity of the 79-1G aggregate fractions. This procedure is a modification of ASTM C 128-59, Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate.

1. Place approximately 500 grams of the fine aggregate fraction, selected by a suitable method of sampling, in an oven at 212°F to 230°F for 24 hours.
2. Determine the weight of a 250 ml. flask. Weight = W_f .
3. Add 250 ± 5 grams of the dried aggregate to the flask and weigh. Weight = W_{fa} .
4. Fill the flask almost to the 250 ml. mark with distilled water. Then roll the flask on a flat surface to remove entrapped air and then apply a vacuum for the same reason.

5. Next place the flask in a constant temperature bath maintained at room temperature.

6. After 24 hours, fill the flask to the 250 ml. mark and determine the weight. Weight = W_{faw} .

7. All weights are to be determined to the nearest 0.01 gram.

8. Record room temperature after step 6.

9. Determine the specific gravity as follows:

$$\begin{aligned} \text{Sp.Gr.} &= \frac{W_{fa} - W_f}{250 - (W_{faw} - W_{fa}) \gamma_w} \\ &= \frac{W_a}{W_{wd} \gamma_w} \end{aligned}$$

$W_a = W_{fa} - W_f$ = weight of aggregate

$W_{wd} = 250 - (W_{faw} - W_{fa})$ = weight of water displaced

γ_w = unit weight of water at the temperature recorded in step 6

It was decided to make three specific gravity determinations for each aggregate fraction. These are listed in Table 23, along with the corresponding data, and the specific surfaces are given in Table 24.

Sample Calculation:

Size fraction No. 8 - No. 16

$W_f = 83.32$, $W_{fa} = 331.66$, $W_{faw} = 489.70$

Temp. = 22.5°C , $\therefore \gamma_w = .998 \text{ gm/cc}$

$W_a = W_{fa} - W_f = 331.66 - 83.32 = 248.34$

$W_w = W_{faw} - W_{fa} = 489.70 - 331.66 = 158.04$

$W_{wd} = 250 - W_w = 250 - 158.04 = 91.96$

$\text{Sp.Gr.} = G_s = \frac{W_a}{W_{wd} \gamma_w} = \frac{248.34}{91.96 \times .998} = 2.71$

TABLE 23

DETERMINATION OF APPARENT SPECIFIC GRAVITIES OF 79-1G AGGREGATE FRACTIONS

Fraction	Sample Number	Temp. °C	γ_w	W_a $W_{fa} - W_f$		W_{faw}	W_w $W_{faw} - W_{fa}$	W_{wd} $250 - W_w$	$\frac{W_a}{W_{wd}}$	Average Specific Gravity
3/8"	1	22.5	0.998	82.45	331.42	490.04	158.62	91.38	2.73	
to	2	22.5	0.998	77.01	327.09	485.10	158.01	91.49	2.72	2.73
No. 4	3	22.5	0.998	73.57	323.29	481.53	158.24	91.76	2.73	
No. 4	1	22.5	0.998	86.26	337.22	494.91	157.69	92.36	2.72	
to	2	22.5	0.998	97.95	347.64	505.86	158.22	91.78	2.73	2.73
No. 8	3	22.5	0.998	87.31	337.52	495.63	158.11	91.89	2.73	
No. 8	1	22.5	0.998	83.32	331.66	489.70	158.04	91.96	2.71	
to	2	22.5	0.998	86.22	337.70	494.67	156.97	93.03	2.71	2.71
No. 16	3	22.5	0.998	86.38	336.43	494.00	157.57	92.43	2.71	
No. 16	1	22.5	0.998	85.09	334.54	491.75	157.21	92.79	2.69	
to	2	22.5	0.998	95.54	345.31	501.58	156.27	93.73	2.67	2.68
No. 30	3	22.5	0.998	87.74	337.47	493.96	156.49	93.51	2.68	
No. 30	1	22.5	0.998	89.60	340.22	495.67	155.45	94.55	2.66	
to	2	22.5	0.998	87.04	336.32	492.92	156.60	93.40	2.67	2.67
No. 50	3	22.5	0.998	91.36	341.27	497.96	156.69	93.31	2.68	
No. 50	1	22.5	0.998	83.17	332.82	489.43	156.61	93.39	2.68	
to	2	22.5	0.998	83.52	333.01	489.30	156.29	93.71	2.67	2.68
No. 100	3	22.5	0.998	96.95	346.76	504.20	157.44	92.56	2.70	
No. 100	1	22.5	0.998	88.69	338.80	492.20	153.40	96.60	2.59	
to	2	22.5	0.998	89.95	340.58	493.28	152.70	97.30	2.58	2.58
No. 200	3	18.0	0.999	83.21	333.66	486.49	152.83	97.17	2.58	
Passed	1	22.5	0.998	83.21	331.71	485.50	153.79	96.21	2.59	
No. 200	2	22.5	0.998	81.15	330.71	484.35	153.64	96.36	2.59	2.59
	3	18.0	0.999	83.52	333.02	486.52	153.50	96.50	2.59	

TABLE 24
DETERMINATION OF SPECIFIC SURFACES OF 79-1G AGGREGATE FRACTIONS

Material Fraction	Average Specific Gravity	Sieve Openings (cm.)*			$\frac{d_G \gamma}{a s w}$	$\frac{6}{d_G \gamma a s w}$	f Shape Factor	Adjusted S.S. (cm. ² /gm.)
		$\frac{d_L}{d_S}$	$\frac{d_S}{d_a}$	$\frac{d_a}{d_s}$				
3/8" to No. 4	2.73	0.951	0.476	0.7135	1.9479	3.08	1.25	3.85
No. 4 to No. 8	2.73	0.476	0.238	0.357	0.97461	6.12	1.25	7.65
No. 8 to No. 16	2.71	0.238	0.119	0.1785	0.48374	12.40	1.25	15.50
No. 16 to No. 30	2.68	0.119	0.0595	0.08925	0.23919	25.08	1.25	31.35
No. 30 to No. 50	2.67	0.0595	0.0297	0.0446	0.11908	50.39	1.25	62.99
No. 50 to No. 100	2.68	0.0297	0.0149	0.0223	0.05976	100.40	1.25	125.50
No. 100 to No. 200	2.58	0.0149	0.0074	0.01115	0.02877	208.55	1.25	260.69
Passed No. 200	2.59	0.0074	.0037**	.00555	0.01437	417.54	1.25	521.93

* From ASTM E 11-61

** No. 400 sieve, an assumption.

APPENDIX B
METHOD OF RANDOMIZATION

The method of randomization of the order of mixing the batches of mortar for each investigation was determined by the use of a table of random numbers (27). The procedure for the selection of the random numbers was as follows:

1. Arbitrarily select one of the pages of tabled values.
2. Without direction, bring a pencil point down on the printed page so as to hit a random digit.
3. Read this digit and the next three to the right.
4. Let the first two of these specify the row and the last two the column.
5. Go to this point in the table of random numbers and read the specified digit and the next one to the right.
If this number were 62 and the only possible numbers of use in the specified problem were 01 to 12, it would be necessary to scan down the column until a suitable number was observed.
6. Repeat the above procedure until the order of mixing is determined for all of the batches.

APPENDIX C

STATISTICAL ANALYSIS FOR INVESTIGATION NO. 1

The model for this analysis is presented in the section titled Results.

		Mixer Type								Totals
		I				II				
		Specific Surface				Specific Surface				
		50	55	60	65	50	55	60	65	
Fineness Modulus	2.40	10.8	10.5	9.7	12.1	12.8	10.7	9.9	12.1	88.6
		T = 43.1				T = 45.5				
	2.70	9.1	9.4	9.2	9.2	10.0	9.6	10.2	9.1	75.8
		T = 36.9				T = 38.9				
	3.00	7.7	9.9	11.8	9.2	7.5	8.8	11.0	8.2	74.1
		T = 38.6				T = 35.5				
Totals		27.6	29.8	30.7	30.5	30.3	29.1	31.1	29.4	238.5
		T = 118.6				T = 119.9				

$$n = 1, N = 24, T = 238.5, \text{ Total Sum of Squares} = 2412.71 = SS_T$$

$$\text{Mean Sum of Squares} = M_{yy} = \frac{(238.5)^2}{24} = 2370.094$$

$$\text{Sum of Squares, Mixer Type} = SS_M = \frac{(118.6)^2 + (119.9)^2}{12} - M_{yy}$$

$$SS_M = 0.070$$

$$\text{Sum of Squares, Specific Surface} = SS_S =$$

$$\frac{(27.6 + 30.3)^2 + (29.8 + 29.1)^2 + (30.7 + 31.1)^2 + (30.5 + 29.4)^2}{6} - M_{yy}$$

$$SS_S = 1.384$$

$$\text{Sum of Squares, Fineness Modulus} = SS_F = \frac{(88.6)^2 + (75.8)^2 + (74.1)^2}{8} - M_{yy}$$

$$SS_F = 15.707$$

$$SS_{M \times S} = \frac{(27.6)^2 + (29.8)^2 + \dots + (29.4)^2}{3} - M_{yy} - SS_M - SS_S$$

$$SS_{M \times S} = 1.455$$

$$SS_{M \times F} = \frac{(43.1)^2 + (45.5)^2 + \dots + (35.5)^2}{4} - M_{yy} - SS_M - SS_F$$

$$SS_{M \times F} = 2.352$$

$$SS_{S \times F} = \frac{(23.6)^2 + (21.2)^2 + \cdots + (17.4)^2}{2} - M_{yy} - SS_M - SS_F$$

$$SS_{S \times F} = 21.110$$

$$SS_{\text{error}} = SS_T - SS_M - SS_S - SS_F - SS_{M \times F} - SS_{S \times F} - M_{yy}$$

$$SS_{\text{error}} = 0.538$$

ANOVA

<u>Source of Variation</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>Significance</u>
μ	1	2370.084	2370.094		
M_i	1	0.070	0.070	0.778	N.S.
F_j	2	15.707	7.854	87.267	**
MF_{ij}	2	2.552	1.176	13.067	**
S_k	3	1.384	0.461	5.122	*
MS_{ik}	3	1.455	0.485	5.389	*
FS_{jk}	6	21.110	3.518	39.089	**
$\epsilon_{m(ijk)}$	6	0.538	0.090		
Totals	24	2412.710			

N.S. = non-significant

* = significant at 5 percent α -level

** = significant at 1 percent α -level

Table F-values:

At $\alpha = 0.01$, $F_{1,6} = 13.7$, $F_{2,6} = 10.9$, $F_{3,6} = 9.78$, $F_{6,6} = 8.47$

At $\alpha = 0.05$, $F_{1,6} = 5.99$, $F_{2,6} = 5.14$, $F_{3,6} = 4.76$, $F_{6,6} = 4.28$

APPENDIX D

STANDARD MORTAR CALCULATIONS

$$\text{Air content, percent by volume} = 100 - W \frac{(182.7 + P)}{(200 + P)}$$

where: W = weight of 400 ml. of mortar in grams and

P = percentage of mixing water, based on weight of cement used.

Batch No. 1: 210 cc. water, flow = 80%

$$\text{Wt. of measure + mortar} = 1462.8 \text{ gms.}$$

$$\text{Wt. of measure} = \underline{675.8}$$

$$W = \text{Wt. of mortar} = 787.0 \text{ gms.}$$

$$P = \frac{210}{350} \times 100 = 60.0\%$$

$$\text{Percent air} = 100 - 787.0 \frac{(182.7 + 60.0)}{(2000 - 4 \times 60.0)}$$

$$\% \text{ Air} = 15.0\%$$

Batch No. 2: 220 cc. water, flow = 86%, % Air = 14.5%.

Batch No. 3: 225 cc. water, flow = 87%, % Air = 14.8%

APPENDIX E

STATISTICAL ANALYSIS FOR INVESTIGATION NO. 4*

The model for this analysis is presented in the section titled Results.

Analysis for gradations 1, 2, 3: F.M. = 2.40, S.S. = 60 cm.²/gm.

Replication	Gradation			Totals
	I	II	III	
1	10.2	9.9	8.2	
2	9.9	8.0	8.0	
3	9.4	8.0	7.2	
T_i	29.5	25.9	23.4	$\sum_{i=1}^3 T_i = T = 78.8$
n_i	3	3	3	$\sum_{i=1}^3 n_i = N = 9$
$\sum_{i=1}^3 Y_{ij}^2$	290.41	226.01	183.08	$\sum_{i=1}^3 \sum_{j=1}^3 Y_{ij}^2 = 699.50 = SS_T$

$$\text{Mean Sum of Squares} = M_{yy} = \frac{(78.8)^2}{9} = 689.938$$

$$\text{Sum of Squares, Gradation} = SS_G = \frac{(29.5)^2 + (25.9)^2 + (23.4)^2}{3} - M_{yy}$$

$$SS_G = 6.269$$

$$SS_{\text{error}} = SS_T - M_{yy} - SS_G$$

$$SS_{\text{error}} = 3.293$$

ANOVA

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F-ratio	Significance
μ	1	689.938	689.938		
T_i	2	6.269	3.135	5.710	*
ϵ_{ij}	6	3.293	0.549	--	
Totals	9	699.500	--		

* = Significant at 5 percent α -level

Table F-values:

$$\text{At } \alpha = 0.01, F_{2,6} = 10.9$$

$$\text{At } \alpha = 0.05, F_{2,6} = 5.14$$

All of the ANOVA tables for this investigation are presented in Table 13.

* The analysis presented here is an example analysis.

APPENDIX F

STATISTICAL ANALYSIS FOR INVESTIGATION NO. 5

The following are the results of the linear regression analysis on Y_1 ,

where: Y_1 = air content, %

X_1 = fineness modulus

X_2 = specific surface, cm^2/gm .

ANOVA

<u>Source</u>	<u>Sum of Squares</u>	<u>Degrees of Freedom</u>
total	47.985779	35
regression	29.201249	3
error	18.784529	32

Variance = 0.58701655

Standard deviation = 0.76617005

Correlation coefficient = 0.78008951

R-squared = 0.60853965

Regression coefficients

Constant + 54.36326

X_1 - 13.73406

X_2 - 0.6975901

$(X_1)(X_2)$ + 0.2032206

Regression equation:

$$Y_1 = 54.36326 - 13.73406(X_1) - 0.6975901(X_2) + 0.2032206(X_1)(X_2)$$

APPENDIX G

STATISTICAL ANALYSIS FOR INVESTIGATION NO. 6

The following are the results of the linear regression analysis on Y_1 ,

where: Y_1 = air content

X_1 = fineness modulus

X_2 = specific surface

ANOVA

<u>Source</u>	<u>Sum of Squares</u>	<u>Degrees of Freedom</u>
total	18.651710	11
regression	16.704779	3
error	1.9469313	8

Variance = 0.24336642

Standard deviation = 0.49332182

Correlation coefficient = 0.94637015

R-squared = 0.89561646

Regression coefficients

Constant + 48.61519

X_1 - 11.46800

X_2 - 0.5719709

$(X_1)(X_2)$ + 0.1363603

Regression equation:

$$Y_1 = 48.61519 - 11.46800(X_1) - 0.5719709(X_2) + 0.1363603(X_1)(X_2)$$

APPENDIX H

STATISTICAL ANALYSIS FOR INVESTIGATION NO. 7

The following are the results of the linear regression analysis on Y_1 ,
 where: Y_1 = air content, %
 X_1 = fineness modulus
 X_2 = specific surface, cm^2/gm .

ANOVA

<u>Source</u>	<u>Sum of Squares</u>	<u>Degrees of Freedom</u>
total	102.23019	35
regression	77.385528	3
error	24.844666	32

Variance = 0.77639583
 Standard deviation = 0.88113327
 Correlation coefficient = 0.87004212
 R-squared = 0.75697330

Regression coefficients

Constant + 16.50663
 X_1 + 2.096219
 X_2 - 0.01579947
 $(X_1)(X_2)$ - 0.08682261

Regression equation:

$$Y_1 = 16.50663 + 2.096219(X_1) - 0.01579947(X_2) - 0.08682261(X_1)(X_2)$$

A "student's" t-test was applied to the differences of the means of the data from this investigation and Investigation No. 5 for each fineness modulus. The following is an example analysis.

F.M. = 2.40:

Gradation Number	Average Air Content		d	d ²
	Invest. No. 5	Invest. No. 7		
1	11.20	10.60	+ 0.60	0.3600
2	9.93	9.60	+ 0.33	0.1089
3	9.83	8.93	+ 0.90	0.8100
4	7.17	<u>6.47</u>	<u>+ 0.70</u>	<u>0.4900</u>
		Totals	+ 2.53	1.7689

$$H_0: \mu_{i5} = \mu_{i7} \quad H_1: \mu_{i5} \neq \mu_{i7}$$

$$\bar{d} = \frac{2.53}{4} = + 0.6325$$

$$s_d = \sqrt{\frac{1.7689 - \frac{(2.53)^2}{4}}{3}} = 0.2371$$

$$t_{3d.f.} = \frac{\bar{d}}{s_d / \sqrt{n}} = \frac{+ 0.6325}{0.2371/2} = 5.3353$$

$$t_{3d.f., \alpha = 0.01} = \pm 5.841$$

$$t_{3d.f., \alpha = 0.05} = \pm 3.182$$

Conclusion: Accept H_0 at $\alpha = 0.01$

Reject H_0 at $\alpha = 0.05$

F.M. = 2.70: $t = 1.3418$, Conclusion: Accept H_0 at $\alpha = 0.05$

F.M. = 3.00: $t = - 1.1046$, Conclusion: Accept H_0 at $\alpha = 0.05$

A "student's" t-test was also performed on the data to determine if the slopes of the prediction equations for the two sets of data were alike. The calculations for these tests will now be presented by the use of an example.

$$t_{n_5+n_7-4 \text{ d.f.}} = \frac{b_5 - b_7}{s_{b_5-b_7}}$$

$$s_{b_5-b_7}^2 = s_E^2 \left[\frac{1}{\sum_{i=1}^{n_5} (x_{5i} - \bar{x}_5)^2} + \frac{1}{\sum_{j=1}^{n_7} (x_{7j} - \bar{x}_7)^2} \right]$$

$$s_E^2 = \frac{\sum_{i=1}^{n_5} (y_{5i} - \hat{y}_{5i})^2 + \sum_{j=1}^{n_7} (y_{7j} - \hat{y}_{7j})^2}{n_5 + n_7 - 4}$$

$$F.M. = 2.40:$$

$$\bar{x}_5 = \bar{x}_7 = 57.5 \quad x_{i5} = x_{i7}$$

$$H_0: b_5 = b_7 \text{ at } F.M. = 2.40, H_1: b_5 \neq b_7 \text{ at } F.M. = 2.40$$

x_{5i}	$(x_{5i} - \bar{x}_5)^2$	y_{5i}	$\hat{y}_{5i}$	$(y_{5i} - \hat{y}_{5i})^2$	y_{7i}	$\hat{y}_{7i}$	$(y_{7i} - \hat{y}_{7i})^2$
50	56.25	11.20	10.91	0.0841	10.60	10.33	.0729
55	6.25	9.93	9.86	0.0049	9.60	9.21	.1521
60	6.25	9.83	8.81	0.0004	8.93	8.09	.7056
65	56.25	7.17	7.76	0.3481	6.47	6.97	.2500
	125.00			0.4375			1.1806

$$s_E^2 = \frac{0.4375 + 1.1806}{4 + 4 - 4} = 0.404525$$

$$s_{b_5-b_7}^2 = 0.404525 \left[\frac{1}{125.00} + \frac{1}{125.00} \right] = 0.0064724$$

$$s_{b_5-b_7} = \sqrt{0.0064724} = 0.08045$$

$$b_5 = \frac{10.908 - 7.761}{50 - 65} = - 0.2098$$

$$b_7 = \frac{10.329 - 6.996}{50 - 65} = - 0.2222$$

$$t = \frac{- 0.2098 - (-0.2222)}{0.08015} = + 0.1541$$

$$t_{4d.f., \alpha = 0.05} = \pm 2.776$$

$$t_{4d.f., \alpha = 0.01} = \pm 4.604$$

Conclusion: Accept H_0 that $b_5 = b_7$ at F.M. = 2.40

F.M. = 2.70: $t = + 0.6806$, Conclusion: Accept H_0 at $\alpha = 0.05$

F.M. = 3.00: $t = + 6.3312$, Conclusion: Reject H_0 at $\alpha = 0.01$,

Accept H_1 : $b_5 \neq b_7$ at F.M. = 3.00

APPENDIX I

STATISTICAL ANALYSIS FOR INVESTIGATION NO. 8

The model for this analysis is presented in the section titled Results.

Replication	Aggregate Size Fractions*				Totals
	I	II	III	IV	
1	9.4	12.4	12.4	7.1	
2	7.8	10.3	14.9	8.0	
3	11.3	13.0	10.7	8.9	
T_i	35.7	35.7	38.0	24.0	$T = 126.2$
n_i	3	3	3	3	$N = 12$
$\sum_{i=1}^3 Y_{ij}^2$	276.89	428.85	490.26	193.62	$\sum_{i=1}^3 \sum_{j=1}^4 Y_{ij} = 1389.620 = SS_T$

Mean Sum of Squares = $M_{yy} = \frac{(126.2)^2}{12} = 1327.203$

Sum of Squares, Size = $SS_S = \frac{(28.5)^2 + (35.7)^2 + (38.0)^2 + (24.0)^2}{3} - M_{yy}$

$SS_S = 41.710$

$SS_{error} = SS_T - M_{yy} - SS_S$

$SS_{error} = 20.707$

ANOVA

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F-ratio	Significance
μ	1	1327.203	1327.203		
T_i	2	41.710	20.855	9.063	**
ϵ_{ij}	9	20.707	2.301		
Totals	12	1389.620	--		

** = Significant at 1 percent α -level.

Table F-values

At $\alpha = 0.01$, $F_{2,9} = 8.02$

At $\alpha = 0.05$, $F_{2,9} = 4.26$

* Aggregate Size Fractions: I = No. 8 - No. 16, II = No. 16 - No. 30, III = No. 30 - No. 50 and IV = No. 50 - No. 100.

Duncan Test on Means

Size	III		II		I		IV
$\bar{Y}_i$	12.67	>	11.90	>	9.50	>	8.00

$$S_{\bar{Y}_i} = \sqrt{\frac{\text{Error Mean Square}}{n_i}} = \sqrt{\frac{2.301}{3}} = 0.8758 = \text{estimated standard error of } \bar{Y}_i$$

At 1 percent level

$$r_{.01}(4, 9 \text{ d.f.}) = 4.91$$

$$r_{.01}(3, 9 \text{ d.f.}) = 4.79$$

$$r_{.01}(2, 9 \text{ d.f.}) = 4.60$$

At 5 percent level

$$r_{.05}(4, 9 \text{ d.f.}) = 3.42$$

$$r_{.05}(3, 9 \text{ d.f.}) = 3.34$$

$$r_{.05}(2, 9 \text{ d.f.}) = 3.20$$

Least Significant Ranges

$$R_4 = r_{.01}(4, 9 \text{ d.f.}) \cdot S_{\bar{Y}_i} = 4.32$$

$$R_3 = r_{.01}(3, 9 \text{ d.f.}) \cdot S_{\bar{Y}_i} = 4.22$$

$$R_2 = r_{.01}(2, 9 \text{ d.f.}) \cdot S_{\bar{Y}_i} = 4.05$$

$$R_4 = r_{.05}(4, 9 \text{ d.f.}) \cdot S_{\bar{Y}_i} = 3.01$$

$$R_3 = r_{.05}(3, 9 \text{ d.f.}) \cdot S_{\bar{Y}_i} = 2.94$$

$$R_2 = r_{.05}(2, 9 \text{ d.f.}) \cdot S_{\bar{Y}_i} = 2.82$$

Tests for differences of means:

Means Tested	Range	LSR* 1 percent	LSR 5 percent	Significance
III and IV	$R = 12.67 - 8.00 = 4.67$	4.32	3.01	*
III and I	$R = 12.67 - 9.50 = 3.17$	4.22	2.94	**
III and II	$R = 12.67 - 11.90 = 0.77$	4.05	2.82	N.S.
II and IV	$R = 11.90 - 8.00 = 3.90$	4.22	2.94	**
II and I	$R = 11.90 - 9.50 = 2.40$	4.05	2.82	N.S.
I and IV	$R = 9.50 - 8.00 = 1.50$	4.05	2.82	N.S.

* LSR = Least Significant Range

Significance: N.S. = non-significant

* = significant at 1 percent α -level

** = significant at 5 percent α -level

APPENDIX J

STATISTICAL ANALYSIS FOR INVESTIGATION NO. 9

The three-way model and corresponding ANOVA table for this investigation are presented in the section titled Results. The following F-values were used to test for significance:

	$F_{2,48}$	$F_{3,48}$	$F_{6,48}$	$F_{9,48}$	$F_{18,48}$
$\alpha = .01$	5.10	4.24	3.22	2.86	2.32
$\alpha = .05$	3.20	2.81	2.30	2.09	1.84

The two-way analysis of variance was performed on the data for each of the size fractions in order to include as much of the data as possible. In this case the following model was used:

$$Y_{ijk} = \mu + A_i + B_j + AB_{ij} + \epsilon_{k(ij)}$$

where

Y_{ijk} represents the k-th observation of treatment ij, $k = 1, 2$

μ represents the mean effect

A_i represents fineness modulus, $i = 1, 2, 3$

B_j represents the percent of the size fraction present in the aggregate gradation, $j = 1, 2, 3, 4, 5, 6$

AB_{ij} represents the interaction between A and B

$\epsilon_{k(ij)}$ represents the random error within the ij cell

The ANOVA tables for this analysis are presented in the section titled Results. The following F-values were used to test for significance:

	$F_{2,18}$	$F_{5,18}$	$F_{10,18}$	$F_{2,12}$	$F_{3,12}$	$F_{6,12}$
$\alpha = 0.01$	6.05	4.28	3.54	6.93	5.95	4.82
$\alpha = .0.05$	3.57	2.79	2.43	3.89	3.49	3.00

A multiple linear regression analysis was performed on the same data as used for the two-way ANOVA's. The resulting regression equations are presented in the section titled Results. The following are the results for size fraction No. 8 - No. 16, where

Y_1 = air content, %

X_1 = fineness modulus

X_2 = percent of size fraction, %

ANOVA

<u>Source</u>	<u>Sum of Squares</u>	<u>Degrees of Freedom</u>
total	161.35660	35
regression	134.84350	3
error	26.513100	32

Variance = 0.82853436

Standard deviation = 0.91023863

Correlation coefficient = 0.91415879

R-squared = 0.83568630

Regression coefficients

Constant + 13.19980

X_1 - 0.5437236

X_2 - 0.4472680

$(X_1)(X_2)$ + 0.1278595

Required probability = 0.999900

Probability for X_1 = 0.370957

Delete X_1

ANOVA

<u>Source</u>	<u>Sum of Squares</u>	<u>Degrees of Freedom</u>
total	161.35660	35
regression	134.64040	2
error	26.716190	33

Variance = 0.80958152

Standard deviation = 0.89976748

Correlation coefficient = 0.91347011

R-squared = 0.83442765

Regression coefficients

Constant + 11.73174

X_2 - 0.4072302

$(X_1)(X_2)$ + 0.1130306

Regression equation:

$$Y_1 = 11.73174 - 0.4072302(X_2) + 0.1130306(X_1)(X_2)$$