DETERMINATION OF CEMENT CONTENT
OF HARDENED SLAG CONCRETE

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DETERMINATION OF CEMENT CONTENT OF HARDENED SLAG CONCRETE

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INFORMATION RETRIEVAL DATA

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ABSTRACT: A chemical method has been developed for determining the cement content of hardened portland cement concrete containing blast furnace slag as the coarse aggregate. The results obtained are generally within ± 0.5 sacks per cubic yard of the known cement content for slag aggregate concrete cured three to seven days. The method is based on analyzing for SO_3 from the cement. The procedure is also applicable to concrete containing gravel and limestone coarse aggregate, but the results are somewhat less accurate than the results for slag aggregate concrete.

KEY WORDS: Cement content, slag coarse aggregate, concrete composition, concrete testing.

INTRODUCTION

The increasing use of concrete containing blast furnace slag coarse aggregate in Michigan highway construction has resulted in several requests for determination of its cement content. Because of the problems discussed in the following paragraphs, a proposal was drafted for submission to the Bureau of Public Roads in February 1967, for a project intended to "...develop a new method for determining the cement content of hardened concrete, particularly concrete containing slag aggregate." In addition to the exploration and development phase, the Objectives section of the Proposal also sought "To define the precision and accuracy of the method, when analyzing concrete samples prepared with cements and aggregates (slag and natural) from various sources."

The standard method, "Cement Content of Hardened Portland Cement Concrete, ASTM C 85" presently in use for natural aggregate concretes, involves determining the soluble calcium oxide or soluble silica content of the concrete--correcting this value with an aggregate blank--and back-calculating to obtain the cement content. This method is not applicable to concrete containing slag aggregate because slag liberates large amounts of both calcium oxide and silica during the procedure, which would mask the same constituents from the cement. The standard method is subject to errors as great as 10 to 20 percent, even when used for cement content analysis of natural aggregate concrete (1).

Other techniques, such as neutron activation analysis (2) and x-ray spectroscopy (3) have been used to determine cement content of hardened concrete. Since the same elements were determined as in the standard chemical method, the same limitations apply. A microscopic technique has also been used which determines the volume relationships of the constituents of the concrete and permits calculation of the cement content (4). This method is more time consuming than chemical methods when a number of samples are involved and also requires a trained, experienced microscopist. The accuracy of the microscopic method is no better than the chemical method except for samples with very low cement content.

The following research procedure was used in this investigation:

1. Samples of various brands of cement and slag aggregates used in Michigan highway construction were obtained and chemically analyzed to select a suitable constituent on which to base cement content. Sulfur trioxide (SO_3) was selected.

2. An experimental design for the analysis of known concrete mixes was developed that would facilitate statistical evaluation of the analytical procedure and the interactions of the various ingredients combined in the concrete mixes.

3. Standard slag aggregate and natural aggregate concrete mixes of known cement content were prepared in the Research Laboratory and, after curing, ground to -150 mesh for chemical analysis.

4. The standard concrete mixes were analyzed for SO_3 content by a gravimetric chemical method. Both types of standard natural aggregate concrete mixes were also analyzed for soluble silica by an ASTM method (C 85-66).

5. The chemical data were converted to cement content and statistically evaluated for component interaction, accuracy, and precision.

PRELIMINARY STUDIES

Selection of a Suitable Constituent

Any method developed for determining the cement content of hardened concrete must be based on some chemical constituent present in the cement, but absent or nearly absent in the aggregate. Comparison of chemical composition data for blast furnace slags and portland cements eliminated silica, calcium oxide, magnesium oxide, iron oxide, aluminum oxide, titanium oxide, and manganese oxide as bases for determining the cement content of concrete containing slag aggregate.

The following table presents percentage composition ranges which will include most blast furnace slags[1] and Type I Portland cements.[2]

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(1) "Iron Blast Furnace Slag; Production, Processing, Properties and Uses," U.S. Bureau of Mines, Bulletin No. 479.

(2) "Design and Control of Concrete Mixtures," Portland Cement Association, Skokie, Illinois, 11th edition p. 16 (1968).

	Blast Furnace Slag	Portland Cement
SiO_2	33-42	19-25
CaO	36-45	60-66
MgO	3-12	0-5
Fe_2O_3	0.3-2	1-5
Al_2O_3	10-16	3-8

Analysis of a sample of blast furnace slag yielded values of 0.3% for both titanium dioxide (TiO_2) and manganese oxide (Mn_2O_3). Corresponding values for a Type I portland cement were, TiO_2 - 0.2%, Mn_2O_3 - 0.05% Analytical values of TiO_2 - 0.27%, Mn_2O_3 - 0.05% are also reported by the National Bureau of Standards for Standard Sample No. 177 of Portland Cement.

Sulfur trioxide (SO_3) content for Type I portland cements from 13 different plants that were used in Michigan over the period 1956 to 1967 ranged from 1.5 to 2.9%. The average SO_3 content was 2.3% with 90% of the samples at or above 2.0% SO_3. The SO_3 contents of four samples of slag from various possible sources ranged only between 0.00 and 0.26%. This difference in SO_3 content was deemed sufficient to provide the basis for an analytical method.

Other Preliminary Studies

Samples of slag from three of four sources were obtained weekly for a six-week period to investigate the variability in SO_3 content of the slags; only two samples of the fourth slag were available during this period. Significant variations in SO_3 content were noted for all slags, indicating the necessity of obtaining samples of the aggregate actually used in a concrete mix in order to achieve an accurate cement content analysis.

The possibility that sulfur compounds in the slag other than sulfates might be oxidized to sulfates during aggregate drying and aging, curing of concrete, or during the drying of ground concrete before analysis, was considered. Such an oxidation process would lead to high SO_3 results and high cement content values. Thus, samples of the four different slags were heated in an oven at 110 C for several hours. Other samples were exposed outdoors for several months. Chemical analysis of the slags before and after such treatment disclosed that only one of the slags changed significantly in SO_3 content (0.09 to 0.14 percent).

Also, two trial concrete mixes (6 sacks/cu yd) were prepared, using two different slags with the same brand of cement, and cast in 4- by 8-in. cylinders to check the effects of curing, grinding, and drying concrete for analysis. One cylinder from each mix was analyzed for SO_3 after curing for 1, 3, 7, and 28 days, respectively.

Minor non-systematic differences in SO_3 content were found at the various cure times. This indicated that later standard mixes should be analyzed at more than one cure time.

As a check on the analytical part of the procedure, free from variability due to concrete mixing, curing, grinding, etc., dry blends of cement, -150 mesh slag aggregate, and -150 mesh sand, in mix design proportions, were analyzed for SO_3. Table 1 presents the analytical data obtained for combinations of four slags and two brands of cement. These data made it apparent that the analytical procedure itself was capable of results well within the specified maximum error of ± 0.50 sacks of cement per cu yd.

EXPERIMENTAL DESIGN

Slag Aggregate Concrete

The experimental design for the slag aggregate concrete phase of the study was planned in detail with the assistance of staff statisticians. The design considered possible component interactions, such as the way a specific cement brand might react in a mix with a certain slag at a specified cement content. The cure time variable was included to determine the effect of the curing process on cement content results. The final design was based on combinations of four slags, two cement brands, and two nominal cement contents (3.0 and 6.0 sacks/cu yd) for a total of 16 mixes. Portions of each mix were to be analyzed at 1, 3, 7, and 28 day cure times. Triplicate samples were scheduled for analysis at each cure time. Thus 192 analyses were scheduled. In addition, some mixes were to be analyzed by a second analyst to evaluate operator variability. Figure 1 shows the experimental design.

TABLE 1
SO_3 CONTENTS OF DRY LABORATORY BLENDS
OF SLAG AGGREGATE CONCRETE INGREDIENTS

Slag Type	Cement Brand	Nom. Mix, sacks/ cu yd	Calculated SO_3 Fraction r	Experimental SO_3 Fraction s	Difference s-r	Difference as Cement Content, sacks/cu yd
A	1	3	0.0022	0.0024	+0.0002	0.34
		6	0.0038	0.0040	+0.0002	0.34
	2	3	0.0020	0.0022	+0.0002	0.38
		6	0.0035	0.0038	+0.0003	0.63
B	1	6	0.0044	0.0042	-0.0002	0.34
	2	3	0.0025	0.0026	+0.0001	0.20
		6	0.0040	0.0042	+0.0002	0.40
C	1	3	0.0017	0.0018	+0.0001	0.17
		6	0.0033	0.0033	0.0000	0.00
	2	3	0.0016	0.0016	0.0000	0.00
		6	0.0031	0.0032	+0.0001	0.20
D	1	3	0.0019	0.0021	+0.0002	0.34
		6	0.0035	0.0036	+0.0001	0.18
	2	3	0.0016	0.0015	-0.0001	0.19
		6	0.0031	0.0030	-0.0001	0.18
					Average	0.26

Slag Type	Cement Brand	Cement Content							
		3 sacks/cu yd				6 sacks/cu yd			
		Curing time, days				Curing time, days			
		1	3	7	28	1	3	7	28
A	1	RRR	RRR	RRR	RRR	RRR	RRR	RRR	RRR
B	1	RRR	RRR	RRR	RRR	RRR	RRR	RRR	RRR
C	1	RRR	RRR	RRR	RRR	RRR	RRR	RRR	RRR
D	1	RRR	RRR	RRR	RRR	RRR	RRR	RRR	RRR
A	2	RRR	RRR	RRR	RRR	RRR	RRR	RRR	RRR
B	2	RRR	RRR	RRR	RRR	RRR	RRR	RRR	RRR
C	2	RRR	RRR	RRR	RRR	RRR	RRR	RRR	RRR
D	2	RRR	RRR	RRR	RRR	RRR	RRR	RRR	RRR

Figure 1. Experimental design for slag concrete cement determination (R = replicate analysis).

Natural Aggregate Concrete

The experimental design for the natural aggregate concrete phase of the study was planned after the data from slag aggregate concrete had been partially evaluated. The consistency of the slag concrete data indicated that fewer standard mixes would be necessary to evaluate the SO_3 method for natural aggregate concretes. The design was based on one cement brand, two limestone aggregates, two gravel aggregates and two cement contents, for a total of eight mixes. Triplicate samples were scheduled for analysis after cure times of 7 and 28 days, for a total of 48 analyses. In addition, the standard natural aggregate concrete mixes were to be analyzed for cement content by a soluble silica procedure described in ASTM method C 85-66 so that the two methods could be compared.

EXPERIMENTAL PROCEDURE

Preparation of Standard Concrete Mixes

Empirically based mix designs, modified from normal highway construction mixes, were used to obtain concrete with satisfactory handling characteristics for this study. The design air content was five percent by volume. Typical mix design data are included in Appendix I. Aggregates were in a water-saturated, surface dry condition. The batch sizes were calculated to include the volume of the samples (eight 4- by 8-in. wax-coated paper cylinder molds), air meter capacity (about 0.1 cu ft), and a 5-percent waste allowance.

The calculated weights of dry ingredients were placed in a 2.25 cu ft electrically driven tilting drum type mixer. The mixer was operated while water was carefully measured into the mix until it was of the desired consistency. The proper amount of Darex[3] air-entraining agent was added with the mix water. After mixing for five minutes, air content was determined on a portion of the mix using a Rollometer for slag concrete and an Acme Pressure Air Meter for the limestone and gravel concrete. The remaining mix was placed in tared cylinder molds and the full molds weighed. The concrete was cured in the cylinder molds on a laboratory bench under polyethylene sheeting for 24 hours, and the molds stripped. The one-day cure specimens were prepared for analysis. The 3-, 7-, and 28-day cure cylinders were stored in a moist room for curing.

Chemical Analysis of Standard Mixes

A. Sample Preparation

Two cylinders were taken at each cure time, air dried, and weighed. Both

(3) Product of Dewey & Almy Chemical Co., Cambridge, Massachusetts.

cylinders were broken in compression and all the pieces saved. One of the broken cylinders was oven-dried at 105 C for 24 hours and reweighed. The other cylinder was broken into 1/4-in. pieces with a jaw crusher, then split with a sample splitter to select a 500 g portion for analysis. This portion was dried in a vacuum oven at 60 C for two hours, then ground to -150 mesh with a 6.5 in. McCool disc pulverizer. The powdered concrete was thoroughly mixed and dried in the vacuum oven for about an hour. Representative portions of the aggregates were similarly prepared.

The weights of the cylinders of fresh concrete, air dry concrete, and oven dry concrete were used to determ ine the water of hydration (bound water) retained at each cure time (See Appendix II for bound water data). The bound water content affects the density of the concrete and the density is needed to convert analytical data to cement content, as illustrated later.

B. Chemical Procedures

The standard slag aggregate and natural aggregate concrete mixes and all mix ingredients were analyzed for SO_3 content. The mix water contained no SO_3 . It should be stressed that the SO_3 content of the cement actually used in the concrete under analysis must be determ ined, due to batch-to-batch variations in the SO_3 content of portland cements. The gravimetric method used (Appendix III) involves extraction of the -150 mesh material with hydrochloric acid, followed by precipitation of sulfate as barium sulfate. Samples of the aggregates used were available in this study, so it was not necessary, as might be the case for field samples, to break down the concrete to obtain aggregates for blank determinations.

The limestone and gravel aggregate concrete mixes were also analyzed by a soluble silica procedure described in ASTM Method C 85-66, "Cement Content of Hardened Portland Cement Concrete." Only the soluble silica procedure from that method was employed. We did not follow the ASTM procedure for breaking down the bulk concrete prior to analysis. The SO_3 method for natural aggregate concrete required roughly half as many man hours and half as much elapsed time as the soluble silica method. A limited number of calcium oxide analyses were also run on gravel aggregate mixes, but the high aggregate blank rendered that method useless.

Calculations

The actual mix volume and the correct cement content for the standard mixes were calculated as in the following example. The mix design shown below is for a slag aggregate mix estimated to yield 0.60 cu ft of concrete with a cement content of 6.0 sacks per cu yd.

Ingredient	Weight, lbs	Volume, cu ft
Cement, Net	12.56	0.0640
Estimated mix water	5.00	0.0800
Fine aggregate (dry sand)	31.63	0.1918
Coarse aggregate (dry slag)	33.94	0.2340
Additional Net Mix Water	1.25	0.0201
TOTAL	84.28	0.5899
Air Content Correction		0.0264
Total Corrected Mix Volume		0.6163

The "additional water" was required in addition to the estimated mix water to produce concrete with the desired consistency. The correct cement content for this mix then, is:

$$\frac{12.56 \text{ lb cement} \times 27.00 \text{ cu ft/cu yd}}{94.00 \text{ lb/sack} \times 0.616 \text{ cu ft}} = 5.85 \text{ sacks cement/cu yd}$$

The SO_3 or soluble silica content of the concrete was converted to cement content as follows:

$$\text{Cement content, sacks/cu yd} = \frac{a - b}{c - b} \times \frac{\text{density of concrete, lb/cu ft} \times 27 \text{ cu ft/cu yd}}{94 \text{ lb/sack}}$$

where:

a = SO_3 or soluble silica fraction in oven dry concrete

b = SO_3 or soluble silica fraction in dry aggregate

c = SO_3 or soluble silica fraction in dry unhydrated cement

Appendix IV provides a detailed description of the derivation of this calculation method and also shows a more rigorous relationship which was simplified to obtain the equation above.

The aggregate blank correction (b) is calculated as in the following example:

For a mix design using coarse and fine aggregate in a 1:1 ratio by weight, with experimental data of

SO_3 fraction of dry coarse aggregate = 0.0019

SO_3 fraction of dry fine aggregate = 0.0001

b = 0.50 x 0.0019 + 0.50 x 0.0001

= 0.0010

RESULTS AND DISCUSSION

Slag Aggregate Concrete

Table 2 gives typical cement content results for standard slag mixes, cured 7 days. Complete data for cure times of 1, 3, 7, and 28 days, and the individual

TABLE 2
CEMENT CONTENT (SACKS/CU YD)
OF STANDARD SLAG AGGREGATE CONCRETE MIXES
MEASURED AFTER 7 DAY CURE

Slag Type	Cement Brand	Nom. Cement Content	Known Cement Content, J	Cement Content, K	Error, J-K	Cement Content, Z Included	Relative Error, percent $\frac{(J-K)}{J} \times 100$
A	1	3 6	2.76 5.78	2.86 5.45	-0.10 +0.33	2.88 5.46	3.6 5.7
	2	3 6	2.84 6.01	2.67 6.12	+0.17 -0.11		6.0 1.8
B	1	6	5.85	5.65	+0.20	5.69	3.4
	2	3 6	2.94 5.80	2.53 5.76	+0.41 +0.04	2.57 5.80	14.0 0.7
C	1	3 6	2.85 5.94	2.82 5.64	+0.03 +0.30		1.1 5.0
	2	3 6	2.78 5.85	2.70 5.61	+0.08 +0.24		2.9 4.1
D	1	3 6	2.88 6.00	2.95 6.47	-0.07 -0.47		2.4 7.8
	2	3 6	2.84 5.75	3.09 5.29	-0.25 +0.46		8.8 8.0

Average Relative Error % 5.0

triplicate analyses, are tabulated in Appendix V. The two different results reported are obtained by: (1) the normal calculation method based on the experimentally determined density of the concrete but neglecting the tiny bound water correction Z (defined in Appendix IV), and (2) for a limited number of samples, calculations based on experimental density and including Z. The very small difference in calculated cement content resulting from inclusion of the Z factor shows that Z can properly be neglected. Table 3 gives results obtained for two mixes prepared some time apart, from the same ingredients, to illustrate the repeatability that might be expected of the method.

TABLE 3
CEMENT CONTENT OF TWO NOMINAL SIX SACKS/CU YD STANDARD CONCRETE MIXES PREPARED FROM THE SAME SLAG TYPE AND CEMENT BRAND[1]

Slag Type	Cement Brand	Known Cement Content, J	Cement Content, K	Error, J-K	Relative Error, percent $\frac{(J-K)}{J} \times 100$
D	2	5.75 sacks/cu yd	5.29	0.46	8.0
		6.14 sacks/cu yd	5.61	0.53	8.6

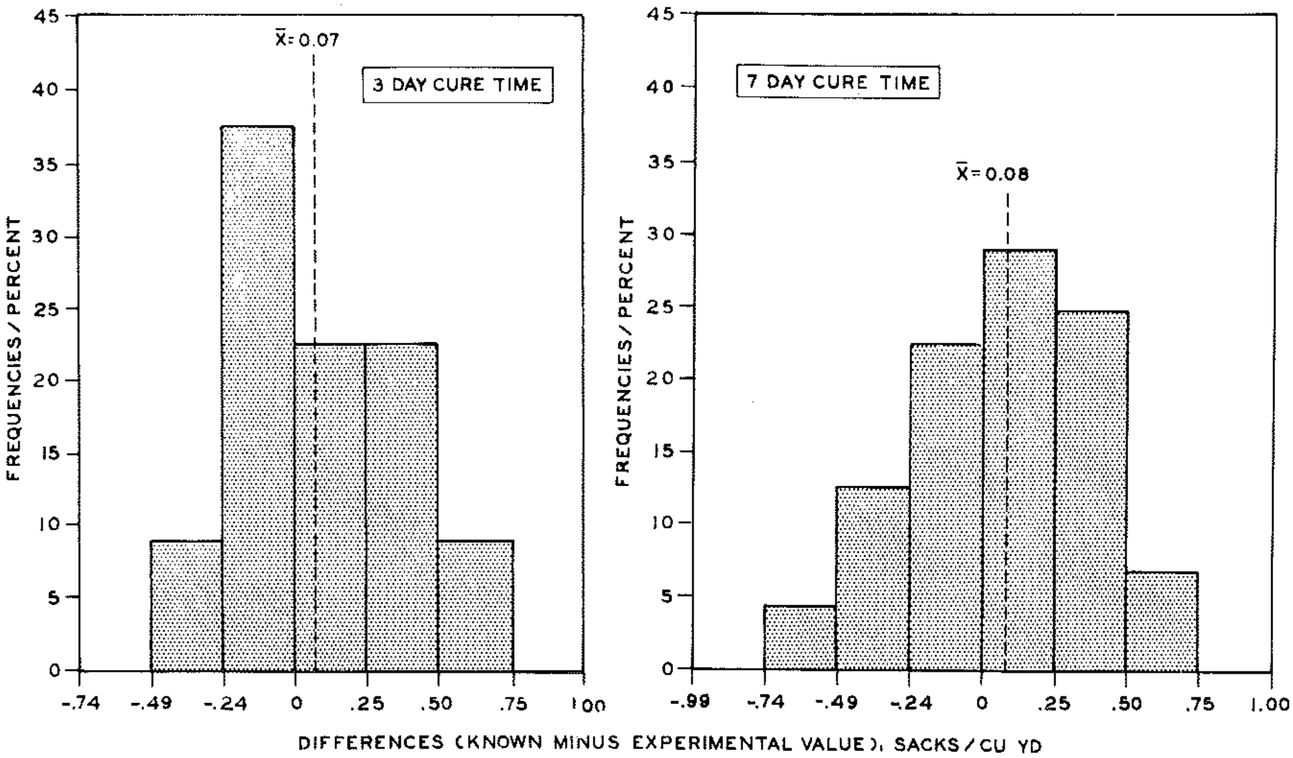
[1] The two mixes were prepared several weeks apart using slightly different mix designs.

The results tended to be low for both calculation methods. There was no significant difference in the overall results obtained by the two operators. Statistical analysis of the data obtained by Operator I yielded the following results:

Cure Time, days	Avg. of 1, 3, 7, & 28	3	7
Mean deviation (known minus experimental)[4], $\bar{x}$, sacks/cu yd	0.10	0.07	0.08
Standard deviation, sacks/cu yd	0.32	0.26	0.28

Figure 2 shows the frequency distribution of results for slag aggregate mixes at various cure times. Results for samples cured 3 days approach the known

(4) $\bar{x}$ is the algebraic sum of deviations from the known cement content divided by the number of measurements.



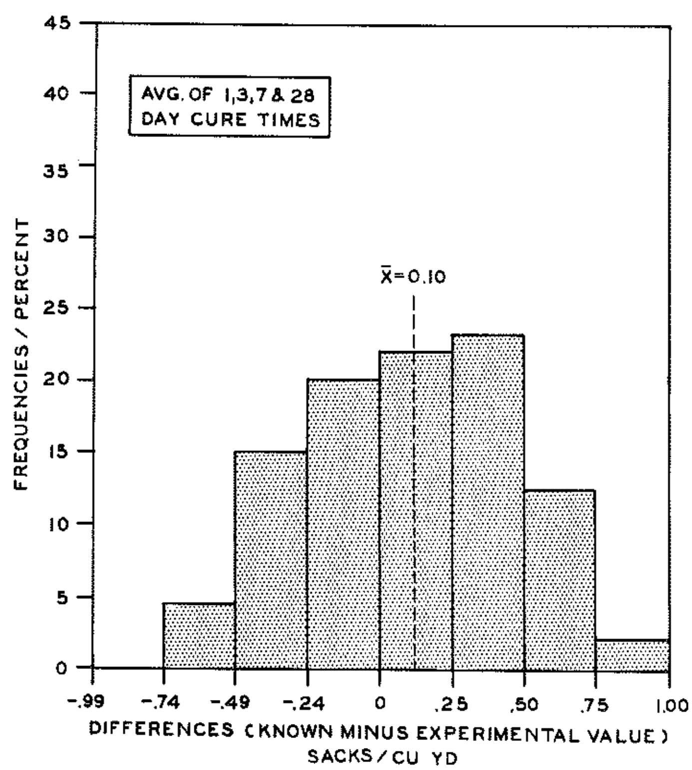


Figure 2. Frequency distributions for slag aggregate mixes at various cure times.

value more closely than those for other cure times. The least accurate results were obtained at very short cure times or at cure times beyond 7 days. For the 3- and 7-day cure results only, 90 percent are within ± 0.50 sacks per cu yd and all of the results are within ± 0.75 sacks per cu yd of the known value. Considering all four cure times, 81 percent of the results are within ± 0.50 sacks per cu yd and 98 percent of the results are within ± 0.75 sacks per cu yd of the known value.

Other detailed analysis of the data for Operator I reveals that the various types of slag and brands of cement interact to significantly affect cement content results. Figure 3 shows the deviation of analytical results for the slag type-cement brand combinations studied. The mean deviations in this and following figures are the algebriac sum of deviations from the known cement content (known minus experimental) divided by the number of measurements.

At a cure time of 28 days, slag type C yields less accurate results with both cements than the other three slags. Also, cement brand 1 generally yields less accurate results than cement brand 2. To guard against a deviation pattern such as that of slag C-cement brand 1, analysis of an unknown concrete containing slag aggregate should be conducted during the approximate cure time interval of 3 to 10 days.

Additional plots of the data present other views of slag type-cement brand interactions. Figure 4 is a composite showing the mean deviation of results for the two brands of cement plotted against slag type. Again, cement brand 1 gives less accurate results than brand 2, and slag C gives the least accurate results of the four slags. Another plot, Figure 5, shows deviation of analytical results for the four slag types averaged over the two brands of cement. Figure 6

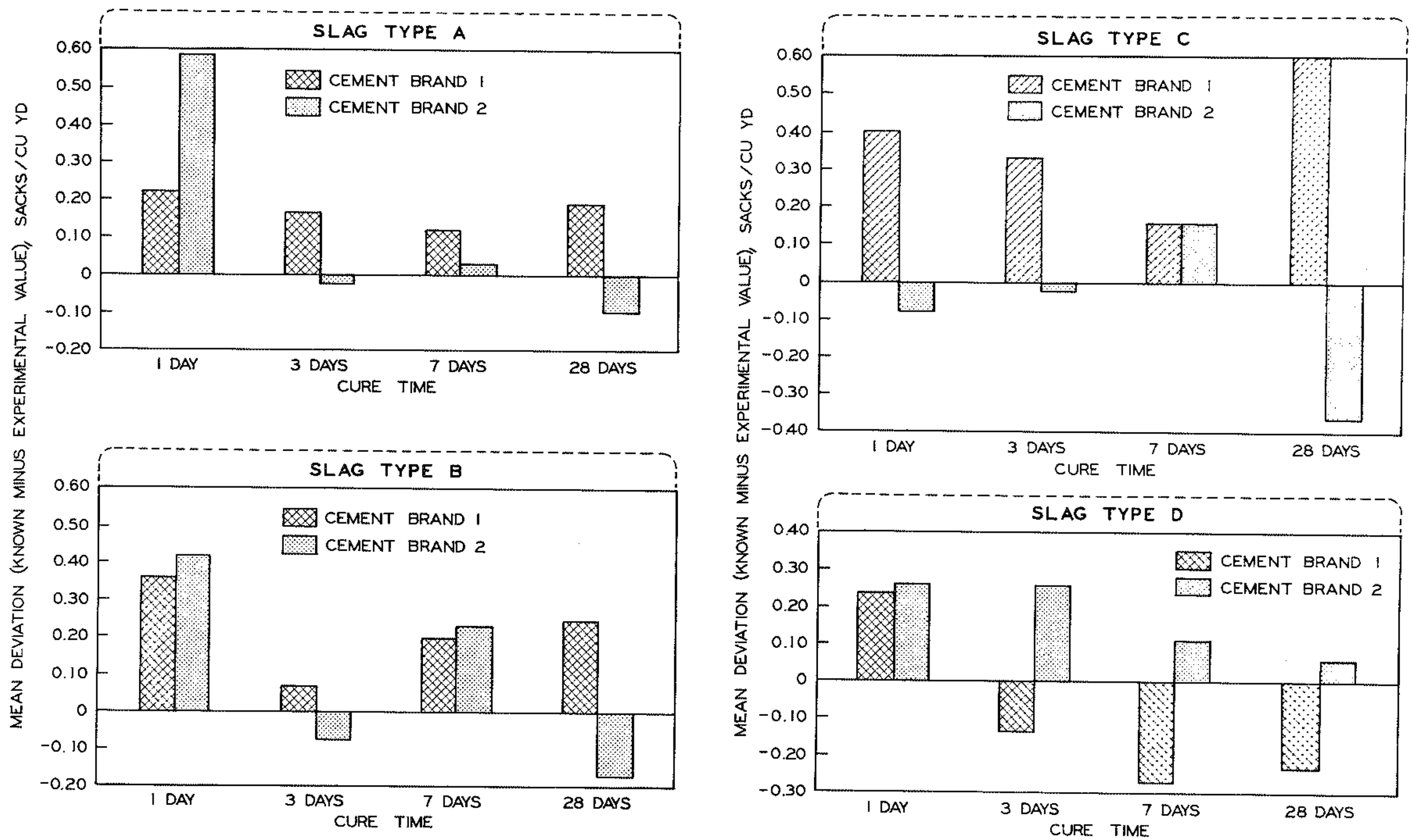


Figure 3. Interaction patterns of the various combinations of slag types and cement brands over the 28-day curing period.

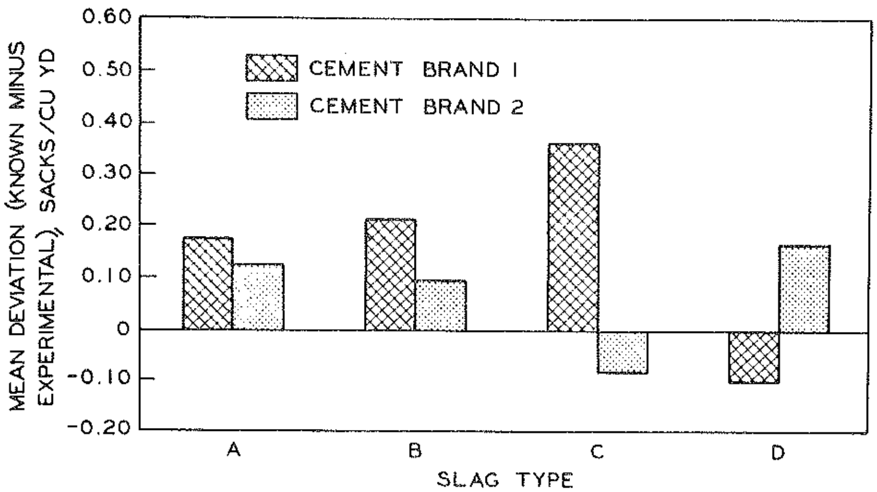
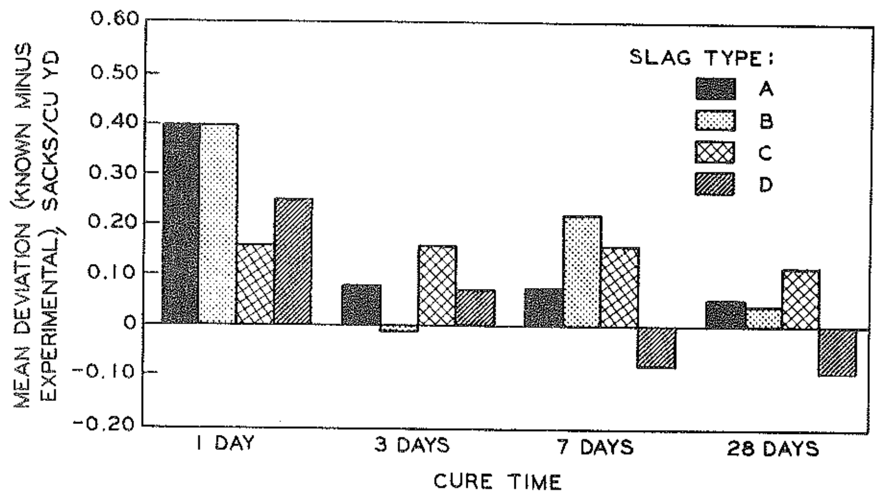


Figure 4. Composite plot of slag type-cement brand interactions, averaged over the four cure times.

Figure 5. Deviation of results as a function of slag type, averaged over two cement brands.



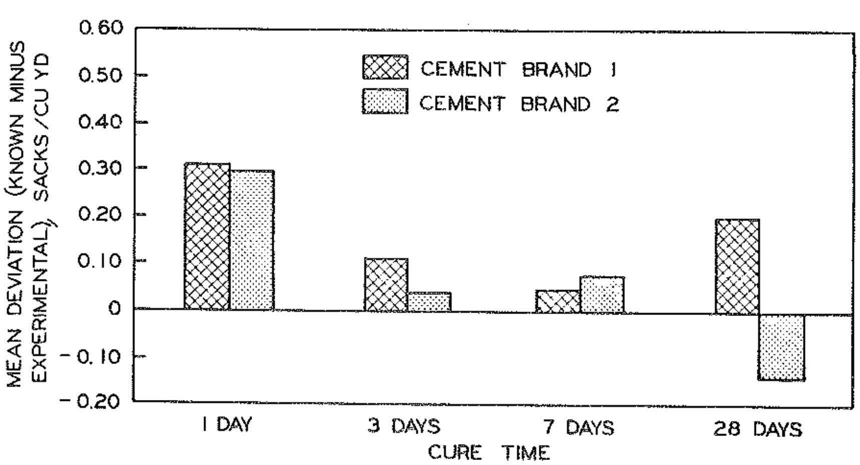


Figure 6. Deviation of results as a function of cement brand, averaged over four slag types.

presents a similar plot of data showing variation of results as a function of cement brand.

Natural Aggregate Mixes

Analysis of the initial 3 and 6 sack per cu yd mixes by both the SO_3 and silica method yielded low results at short cure times, and even lower results were obtained at longer cure times. It was concluded that problems in preparing the samples invalidated the results. A second series of two standard limestone and two standard gravel aggregate concrete mixes (6 sacks/cu yd only) were prepared and placed in 6- by 12-in. paper cylinder molds. Two cylinders were prepared for each mix, for analysis after curing 7 and 28 days, respectively. These cylinders were cured in sealed polyethylene bags containing a beaker of water to maintain high relative humidity.

To obtain additional information about the effects of sample preparation, the 7-day cure cylinders were broken in compression and divided into halves. One half was crushed and ground for analysis. The other half, in the form of large chunks, was returned to a polyethylene bag to complete a 28-day cure period. Cylinders so treated were designated 7-28 day samples.

Analysis of the 7-, 7-28-, and 28-day cure cylinders yielded acceptable results for the limestone mixes, but unacceptable results for the gravel mixes. As can be seen in Appendix VI, the 7-28 day samples indicated there was variability due to sample preparation, even for two portions of the same cylinder. Larger samples of powdered concrete were taken for analysis in this series, because the absence of SO_3 in the aggregate lowered the yield of barium sulfate in the analytical step.

To conclude this phase of the study, a third series consisting of two standard gravel aggregate mixes (6 sacks/cu yd) were prepared under rigorous supervision. The fresh concrete was molded into 6- by 12-in. cylinders which were cured in polyethylene bags for 7 and 28 days, respectively. The 7-day cylinder was not divided for further cure.

Averaged analytical results for the second series of limestone aggregate mixes and the third series of gravel aggregate mixes by the SO_3 and soluble silica methods are given in Table 4. Complete analytical data, by both methods, for the second and third series of natural aggregate mixes may be found in Appendix VI. As in the analysis of slag aggregate mixes, the cement content results for both methods tended to be low and there was no significant variability between two operators. Cure time had no consistent effect on the results for either method over the 7 to 28 day period studied. Analytical results for statistical comparison of the two methods were extracted from Appendix VI (See Appendix VII). The SO_3 method has a mean deviation from the known cement content of 0.33 sacks per cu yd, and a standard deviation of 0.33 sacks per cu yd. The soluble silica method has slightly better accuracy, but somewhat poorer precision than the SO_3 method, as indicated by a mean deviation from the known cement content of 0.30 sacks per cu yd, and a standard deviation of 0.43 sacks per cu yd. Figure 7 shows histograms for these data.

The histograms show that 82 percent of the results are within $\pm$ 0.60 sacks per cu yd of the known value and that all of the SO_3 method results are within the range from 0.60 sacks per cu yd above the known value to 0.80 sacks per cu yd below the known value. Comparison values for the silica method are, 74 percent of the results within $\pm$ 0.60 sacks per cu yd and 91 percent of the

TABLE 4

CEMENT CONTENT DATA FOR THE STANDARD NATURAL AGGREGATE CONCRETE MIXES BY BOTH THE SO_3 AND THE SOLUBLE SILICA METHODS

Aggregate	Known Cement Content, J	SO_3 Method					Silica Method				
		7 Day Cure		28 Day Cure		Relative Error, percent $Avg \frac{(J-K)}{J} \times 100$	7 Day Cure		28 Day Cure		Relative Error, percent $Avg \frac{(J-K)}{J} \times 100$
		Cement Content, K	Error, J-K	Cement Content, K	Error, J-K		Cement Content, K	Error, J-K	Cement Content, K	Error, J-K	
Gravel A Series 3	5.98	5.64	+0.34	6.32	-0.34	5.7	5.44	+0.54	5.94	+0.04	4.8
Gravel B Series 3	5.94	5.29	+0.65	5.58	+0.36	8.5	5.98	-0.04	5.79	+0.15	1.6
Limestone A Series 2	6.01	5.38	+0.63	5.82	+0.19	6.8	5.49	+0.52	6.14	-0.13	5.4
Limestone B Series 2	5.86	5.47	+0.39	5.23	+0.63	8.7	5.40	+0.46	5.15	+0.71	10.0

Average Relative Error 7.4

Average Relative Error 5.4

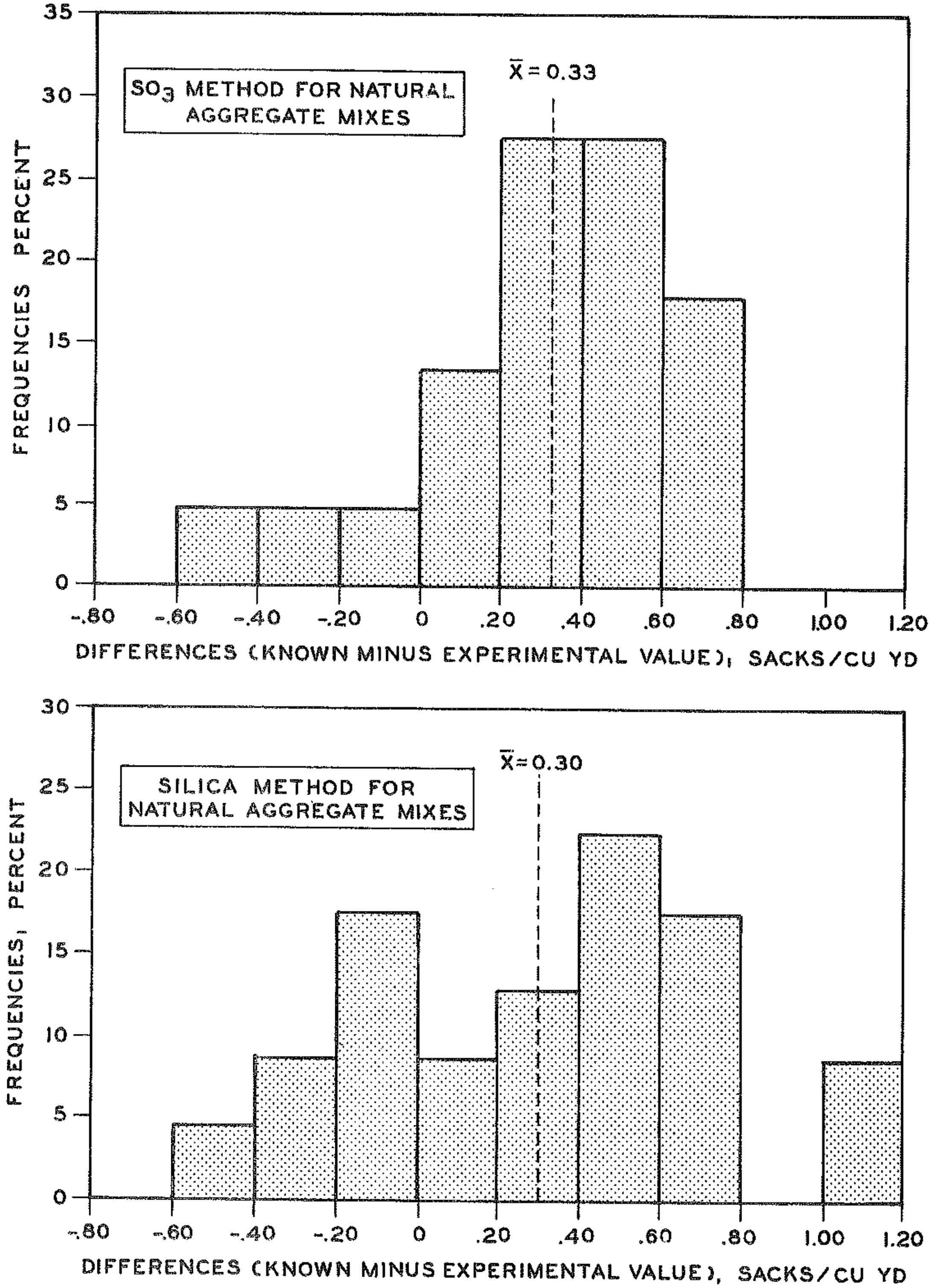


Figure 7. Frequency histograms for cement content results for natural aggregate mixes by the SO_3 and soluble silica methods. The vertical dashed line indicates mean deviation from the known cement content $\overline{X}$.

results within the range from 0.60 sacks per cu yd above the known value to 0.80 sacks per cu yd below the known value.

SUMMARY AND CONCLUSIONS

A satisfactory method for determining cement content of hardened concrete containing blast furnace slag aggregate was developed. The method is also applicable to concrete containing natural aggregates. It requires about half the elapsed time and actual man hours as the soluble silica method (similar to ASTM C 85-66) previously used for natural aggregate samples.

Results obtained by the SO_3 method tended to be low for both natural and slag aggregate concrete. The soluble silica method also tended toward low results for natural aggregate concrete. There was no significant difference in the results obtained by two different operators for either method. For the SO_3 method, 90 percent of the results obtained on slag aggregate concrete samples of nominally 3 and 6 sacks/cu yd were within $\pm$ 0.50 sacks per cu yd of the known value for a curing period of 3 to 7 days. All of the 3 to 7 day cure results were within $\pm$ 0.75 sacks per cu yd of the known value. Less accurate results were obtained at longer or shorter cure times.

For natural aggregate concrete (nominal 6 sacks/cu yd only) 82 percent of the results by the SO_3 method were within $\pm$ 0.60 sacks per cu yd of the known value for a curing period of 7 to 28 days. All of the SO_3 method results for natural aggregate concrete were within the range from 0.60 sacks per cu yd above the known value to 0.80 sacks per cu yd below the known value. Cure time had little effect on the results for natural aggregate concrete by either method. The soluble silica method had 74 percent of the results within ± 0.60 sacks per cu yd of the known value and 91 percent of the results were within the range from 0.60 sacks per cu yd above the known value to 0.80 sacks per cu yd below the known value.

Statistical analysis of the data produced the following values of mean deviation (known minus experimental value), $\bar{x}$, and standard deviation, s.

	SO_3 method			SiO_2 method
	Slag Agg. Avg. 1, 3, 7, 28 day cure	Slag Agg. Avg. 3 and 7 day cure	Natural Agg. Avg. 7 and 28 day cure	Natural Agg. Avg. 7 and 28 day cure
$\bar{x}$, sacks/cu yd	0.10	0.08	0.33	0.30
s, sacks/cu yd	0.32	0.27	0.33	0.43

For slag aggregate concrete, interaction of the different cement brands and types of slag were found to markedly affect the precision and accuracy of cement content results. One brand of cement yielded less accurate results with three of the four slags than the other brand. And, while all the slags had characteristic patterns of cement content results, one type yielded more deviant results than the other three. Half of the mean deviations from the known cement content at a cure time of one day were greater than 0.30 sacks per cu yd but, with the exception of one slag, mean deviations were less than 0.30 sacks per cu yd at cure times longer than three days. In view of this, it is recommended that analysis of an unknown sample of slag aggregate concrete should be carried out during the approximate cure time interval of 3 to 10 days.

It should be emphasized that analytical results for the ingredients actually used in the concrete sample in question must be used in the calculation step to achieve accurate cement content determinations. It was found during the preliminary phases of this study that blast furnace slags from four sources varied significantly in SO_3 content when checked on a weekly basis. Portland Cement also varies in SO_3 content from lot to lot for the same brand due to the empirical addition of gypsum to adjust the time of set within specification limits.

Samples of coarse aggregate for a blank determination could be obtained by crushing part of the hardened concrete sample and picking out pieces of the coarse aggregate. If none of the cement used in a concrete sample is available, the producer might furnish the SO_3 content for the lot in question. Sand or gravel fine aggregate would normally be very low in SO_3 content and samples from the same source would probably have the least variability in SO_3 content of any of the ingredients.

REFERENCES

1. Ford, C L., "A Study of Methods for the Determination of the Portland Cement Content of Hardened Concrete," ASTM Bulletin, No. 181, p. 47, 1952.

2. Covault, D. O., and Poovey, C. E., "Use of Neutron Activation to Determine Cement Content of Portland Cem ent Concrete," Highway Research Board Bulletin No. 340, pp. 1-29, 1962.

3. Mander, J. E., and MacIver, D. Y., "Determination of Cement in Hardened Concrete by X-Ray Spectrography," Pit & Quarry, Vol. 58, pp. 127-8, October 1965.

4. Polivka, M., Kelly, J. W., and Best, C. H., "A Physical Method for Determining the Composition of Hardened Concrete," ASTM Special Technical Publication No. 205, pp. 135-152, 1956.

5. ASTM Special Technical Publication No. 169A, pp. 326-339, 1966.

APPENDIX I

PROPORTIONING FOR TYPICAL STANDARD MIXES[1]

Slag Coarse Aggregate				
	3 sacks/cu yd		6 sacks/cu yd	
	Wt (lbs.)	Vol. (cu ft)	Wt (lbs.)	Vol. (cu ft)
Cement, Type I	6.28	0.0320	12.56	0.0640
Water, Net	6.76	0.1080	6.25	0.1001
Fine Aggregate, dry	38.56	0.2330	31.63	0.1918
Coarse Aggregate, dry	33.81	0.2330	33.94	0.2340
Air		0.0280 (4.4 %)		0.0264 (4.3 %)
Darex		2.0 ml		3.1 ml

Limestone Coarse Aggregate		
	6 sacks/cu yd	
	Wt (lbs.)	Vol. (cu ft)
Cement, Type I	13.63	0.0693
Water, Net	6.00	0.0962
Fine Aggregate, dry	33.25	0.2034
Coarse Aggregate, dry	40.69	0.2489
Air		0.0501 (7.5 %)
Darex		6.4 ml

Gravel Coarse Aggregate		
	6 sacks/cu yd	
	Wt (lbs.)	Vol. (cu ft)
Cement, Type I	13.63	0.0690
Water, Net	6.00	0.0960
Fine Aggregate, dry	29.63	0.1810
Coarse Aggregate, dry	45.75	0.2720
Air		0.0420 (6.3 %)
Darex		6.4 ml

1 The fine aggregate used in all mixes was 2NS gradation sand. Coarse aggregates were graded 6A (1 in. max.). These gradations are detailed in "Standard Specifications, Michigan Department of State Highways, 1967.

APPENDIX II

NON-EVAPORABLE WATER --AS PERCENT OF MIX WATER FOR NATURAL AGGREGATE AND SLAG AGGREGATE MIXES

	Material	Nominal Cement Content, sacks/cu yd	Cure Time, days			
			1	3	7	28
SLAG AGGREGATE	Slag A	3	17.8	21.8	32.7	25.9
		6	18.7	19.4	20.8	31.6
		3	7.4	20.2	30.3	29.4
		6	27.6	38.9	38.8	47.4
	Slag B	6	6.5	18.3	27.6	31.9
		3	22.6	32.5	29.8	32.0
		6	30.4	33.5	35.4	36.2
	Slag C	3	15.8	26.8	28.4	33.3
		6	31.6	33.9	42.6	47.6
		3	20.2	26.0	27.8	21.7
		6	32.3	36.1	37.2	42.0
	Slag D	6	20.3	37.0	37.5	40.9
		3	17.4	23.0	20.4	26.1
		6	33.2	43.4	43.1	47.7
	Average (6 sack mixes)		25.1	32.6	35.4	40.7
NATURAL AGGREGATE	Gravel A	6			27.8	33.1
	Gravel B	6			40.4	39.2
	Limestone A	6			37.1	39.2
	Limestone B	6			46.2	40.5
				Average	37.9	38.0

APPENDIX III

PROCEDURE FOR DETERMINING SO_3 IN HARDENED CONCRETE

1. Crush the standard concrete cylinders to about 1/4-in. pieces, split out about a 500 g sample, dry in a vacuum oven at 60 C for about 2 hours, and grind to pass a 150-mesh sieve. Take care not to lose the fine fractions during crushing and grinding. Further dry the powdered sample for about 1 hour in the vacuum oven and mix it thoroughly to assure homogeneity.

2. Weigh an appropriate sample into a 400 ml beaker. Approximate sample weights are - 5 g for slag concrete mixes, 5 g for straight slag (blank), 10 g for natural aggregate mixes, and 10 g for natural aggregate blanks. Pour 125 ml of water on the sample and, while stirring vigorously, add 25 ml of concentrated HCl. Warm the resulting mixture on a hot plate and break up any lumps with the flattened end of a glass stirring rod until dispersion of the sample is complete.

3. Dilute the mixture to 250 ml, digest for 15 min. just below boiling, then filter through medium fast paper into a 600 ml beaker and wash the residue thoroughly with hot water.

4. Dilute the filtrate to a volume near 600 ml and heat to boiling. Slowly add, drop wise from a pipet, 15 ml of 10 percent barium chloride solution. Continue boiling until the precipitate is well formed (about 1/2 hour). Digest the precipitate for 12 to 24 hours at a temperature slightly below boiling, but maintain the volume above 500 ml.

5. Filter the hot solution through a tared gooch crucible (previously ignited at 800 C) and wash the precipitate with hot water.

6. Ignite the precipitate at 800-900 C, cool in a desiccator, and weigh as $BaSO_4$. Calculations: $SO_3 \text{ fraction} = \frac{W_b \times 0.343}{Ws}$

where:

W_b = weight of $BaSO_4$

0.343 = molecular ratio of SO_3 to $BaSO_4$

Ws = weight of concrete sample

APPENDIX IV

DERIVATION OF CEMENT CONTENT CALCULATION

Let: P = dry unhydrated cement fraction in oven dry concrete

Y = dry aggregate fraction in oven dry concrete

Z = bound water fraction in oven dry concrete

Then, $P + Y + Z = 1$ and $Y = 1 - P - Z$ .... (1)

Now let: a = fraction of analytical constituents, SO_3, in oven dry concrete

b = fraction of analytical constituent in dry aggregate (blank)

c = fraction of analytical constituent in dry, unhydrated cement

These values are readily determined experimentally.

Intuitively, the cement content of the dry concrete is equal to the cement equivalent of the total soluble SO_3 found, minus the cement equivalent of SO_3 extracted from the aggregates. Thus:

$$P = \frac{a - bY}{c} \qquad \text{.................. (2)}$$

and substituting for Y,

$$P = \frac{a - b\,(1 - P - Z)}{c} = \frac{a - b\,(1 - Z)}{c} + \frac{bP}{c}$$

$$P\,(1 - \frac{b}{c}) = \frac{a - b\,(1 - Z)}{c}$$

$$P = \frac{a - b\,(1 - Z)}{c - b} \qquad \text{.................. (3)}$$

Since b and Z are small quantities, their product can be neglected and equation (3) reduced to the following simple form for general use:

$$P = \frac{a - b}{c - b}$$

Values for other analytical constituents, such as soluble silica, can be used in this same equation.

To convert this weight fraction of cement to cement content in sacks per cu yd, use the following relationship:

$$\text{Cement content, sacks/cu yd} = \frac{\text{P (density of oven dry concrete, lb/cu ft) (27 cu ft/cu yd)}}{\text{94 lb/sack of cement}}$$

$$= \text{P multiplied by a constant for the mix}$$

If an experimentally determined density is not available, the density of the oven dry sample can be approximated from the mix design.

For example:

Cement	94.0 lb	
Fine aggregate	237.0 lb	
Coarse aggregate	254.0 lb	
Bound water	15.6 lb	(1/3 of mix water)
Total	600.6 lb/sack	

$$\text{and density, lb/cu ft} = \frac{\text{wt of oven dry concrete}}{\text{sack of cement}} \times \frac{\text{no. of sacks}}{\text{cu yd}} \times \frac{\text{1 cu yd}}{\text{27 cu ft}}$$

$$= \frac{600.6 \times 5.85}{27}$$

$$= 130.1$$

The actual density of this mix was 133.7 lb/cu ft after curing for seven days. As a rough average, this study found that 1/3 of the mix water in slag aggregate concrete is non-evaporable on oven drying for six sack mixes, cured three to seven days.

APPENDIX V (Part 1)

CEMENT CONTENT DATA IN SACKS/CU YD FOR STANDARD SLAG AGGREGATE CONCRETE MIXES, CALCULATED WITH & WITHOUT WATER OF HYDRATION (Z) CORRECTION

Slag Type	Cement Brand	Nominal Cement Content	Known Cement Content J	Operator	One Day Cure Time: Cement Content, K	Error, J-K	Cement Content, Z Included	Error	Three Day Cure Time: Cement Content, K	Error, J-K	Cement Content, Z Included	Error	Seven Day Cure Time: Cement Content, K	Error, J-K	Cement Content, Z Included	Error	Twenty-eight Day Cure Time: Cement Content, K	Error, J-K	Cement Content, Z Included	Error	Average Relative Error, Percent $\frac{Avg\ (J-K)}{J} \times 100$
A	1	3	2.76	I	2.73	+0.03	2.75	+0.01	2.84	-0.08	2.86	-0.10	2.86	-0.10	2.88	-0.12	3.13	-0.37	3.15	-0.39	5.3
				II													3.30	-0.54	3.32	-0.56	
		6	5.78	I	5.38	+0.40	5.40	+0.38	5.36	+0.42	5.38	+0.40	5.45	+0.33	5.46	+0.32	5.03	+0.75	5.05	+0.73	8.2
				II													5.45	+0.33	5.46	+0.32	
	2	3	2.84	I	2.38	+0.46			2.96	-0.12			2.67	+0.17			3.11	-0.27			9.0
				II									3.41	-0.57							
		6	6.01	I	5.39	+0.62			5.93	+0.08			6.12	-0.11			5.93	+0.08			3.7
				II	6.02	-0.01			6.42	-0.41											
B	1	6	5.85	I	5.49	+0.36	5.51	+0.36	5.78	+0.07	5.79	+0.06	5.65	+0.20	5.69	+0.16	5.60	+0.25	5.63	+0.22	3.8
				II					5.90	-0.05	5.92	-0.07									
	2	3	2.94	I	2.50	+0.44	2.52	+0.42	2.99	-0.05	3.02	-0.08	2.53	+0.41	2.57	+0.37	3.12	-0.18	3.17	-0.23	9.2
				II									3.15	-0.21	3.19	-0.25					
		6	5.80	I	5.41	+0.39	5.45	+0.35	5.90	-0.10	5.94	-0.14	5.76	+0.04	5.80	0.00	5.96	-0.16	6.03	-0.23	3.0
				II	6.04	-0.24	6.08	-0.28									6.53	-0.73	6.59	-0.79	
C	1	3	2.85	I	2.32	+0.53			2.46	+0.39			2.82	+0.03			2.33	+0.52			12.9
				II	2.66	+0.19											2.40	+0.45			
		6	5.94	I	5.67	+0.27			5.67	+0.27			5.64	+0.30			5.26	+0.68			6.4
				II													5.38	+0.56			
	2	3	2.78	I	2.62	+0.16			2.82	-0.04			2.70	+0.08			3.27	-0.49			6.9
		6	5.85	I	6.16	-0.31			5.85	0.00			5.61	+0.24			6.14	-0.29			3.6
				II									5.47	+0.38							
D	1	3	2.88	I	2.64	+0.24			3.07	-0.19			2.95	-0.07			2.96	-0.08			5.0
				II									3.59	-0.71							
		6	6.00	I	----	----			6.06	-0.06			6.47	-0.47			6.37	-0.37			5.0
				II					6.75	-0.75											
	2	3	2.84	I	2.79	+0.05			2.97	-0.13			3.09	-0.25			3.03	-0.19			5.5
		6	5.75	I	5.28	+0.47			5.10	+0.65			5.29	+0.46			5.50	+0.25			8.0
				II	5.28	+0.47			5.24	+0.51			5.22	+0.53							

Overall Average 6.4

APPENDIX V (Part 2)

RAW ANALYTICAL DATA FOR STANDARD CONCRETE MIXES CONTAINING SLAG AGGREGATE. CEMENT CONTENT UNITS ARE SACKS/CU YD, CALCULATED NEGLECTING Z-CORRECTION DEFINED IN APPENDIX IV

Slag Type	Cement Brand	Nominal Cement Content	Known Cement Content	Operator	One Day Cure Time			Three Day Cure Time			Seven Day Cure Time			Twenty-eight Day Cure Time		
					SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content
A	1	3	2.76	I	0.0021	2.73		0.0022	2.90		0.0022	2.92		0.0024	3.24	
					0.0021	2.73	2.73	0.0022	2.90	2.84	0.0021	2.75	2.86	0.0023	3.07	3.13
					0.0021	2.73		0.0021	2.73		0.0022	2.92		0.0023	3.07	
				II										0.0024	3.24	
														0.0025	3.42	3.30
														0.0024	3.24	
		6	5.78	I	0.0035	5.26		0.0036	5.36		0.0035	5.33		0.0034	5.15	
					0.0036	5.44	5.38	0.0036	5.36	5.36	0.0036	5.51	5.45	0.0033	4.97	5.03
					0.0036	5.44		0.0036	5.36		0.0036	5.51		0.0033	4.97	
				II										0.0036	5.51	
														0.0035	5.33	5.45
														0.0036	5.51	
A	2	3	2.84	I	0.0017	2.45		0.0020	3.03		0.0019	2.87		0.0021	3.31	
					0.0017	2.45	2.38	0.0020	3.03	2.96	0.0016	2.26	2.67	0.0020	3.11	3.11
					0.0016	2.25		0.0019	2.83		0.0019	2.87		0.0019	2.90	
				II							0.0021	3.28				
											0.0022	3.48	3.41			
											0.0022	3.48				
		6	6.01	I	0.0031	5.39		0.0034	6.07		0.0035	6.26		0.0033	5.88	
					0.0031	5.39	5.39	0.0033	5.86	5.93	0.0034	6.05	6.12	0.0033	5.88	5.93
					0.0031	5.39		0.0033	5.86		0.0034	6.05		0.0034	6.04	
				II	0.0034	6.02		0.0036	6.49							
					0.0034	6.02	6.02	0.0036	6.49	6.42						
								0.0035	6.28							

APPENDIX V (Part 2 Cont.)

RAW ANALYTICAL DATA FOR STANDARD CONCRETE MIXES CONTAINING SLAG AGGREGATE. CEMENT CONTENT UNITS ARE SACKS/CU YD, CALCULATED NEGLECTING Z-CORRECTION DEFINED IN APPENDIX IV

Slag Type	Cement Brand	Nominal Cement Content	Known Cement Content	Operator	One Day Cure Time			Three Day Cure Time			Seven Day Cure Time			Twenty-eight Day Cure Time		
					SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content
B	1	6	5.85	I	0.0041	5.55		0.0041	5.60		0.0042	5.65		0.0041	5.60	
					0.0041	5.37	5.49	0.0041	5.60	5.78	0.0042	5.65	5.65	0.0041	5.60	5.60
					0.0040	5.55		0.0044	6.14		0.0042	5.65				
				II				0.0042	5.78							
								0.0043	5.96	5.90						
								0.0043	5.96							
B	2	3	2.94	I	0.0023	2.57		0.0025	3.05		0.0023	2.60		0.0026	3.19	
					0.0023	2.57	2.50	0.0025	3.05	2.98	0.0023	2.60	2.53	0.0025	2.99	3.12
					0.0022	2.37		0.0024	2.84		0.0022	2.39		0.0026	3.19	
				II							0.0025	3.01				
											0.0026	3.22	3.15			
											0.0026	3.22				
		6	5.80	I	0.0036	5.41		0.0039	6.04		0.0038	5.83		0.0039	6.03	
					0.0036	5.41	5.41	0.0038	5.83	5.90	0.0037	5.62	5.76	0.0038	5.82	5.96
					0.0036	5.41		0.0038	5.83		0.0038	5.83		0.0039	6.03	
				II	0.0039	6.04								0.0041	6.46	
					0.0039	6.04	6.04							0.0041	6.46	6.53
					0.0039	6.04								0.0042	6.67	

APPENDIX V (Part 2 Cont.)

RAW ANALYTICAL DATA FOR STANDARD CONCRETE MIXES CONTAINING SLAG AGGREGATE. CEMENT CONTENT UNITS ARE SACKS/CU YD, CALCULATED NEGLECTING Z-CORRECTION DEFINED IN APPENDIX IV

Slag Type	Cement Brand	Nominal Cement Content	Known Cement Content	Operator	One Day Cure Time			Three Day Cure Time			Seven Day Cure Time			Twenty-eight Day Cure Time		
					SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content
C	1	3	2.85	I	0.0013	2.15		0.0015	2.52		0.0015	2.53		0.0014	2.39	
					0.0015	2.49	2.32	0.0014	2.35	2.46	0.0016	2.71	2.82	0.0013	2.22	2.33
					0.0014	2.32		0.0015	2.52		0.0019	3.23		0.0014	2.39	
				II	0.0015	2.49								0.0016	2.75	
					0.0016	2.66	2.66							0.0013	2.22	2.40
					0.0017	2.83								0.0013	2.22	
		6	5.94	I	0.0031	5.67		0.0031	5.67		0.0030	5.52		0.0029	5.32	
					0.0031	5.67	5.67	0.0031	5.67	5.67	0.0031	5.70	5.64	0.0028	5.14	5.26
					0.0031	5.67		0.0031	5.67		0.0031	5.70		0.0029	5.32	
				II										0.0029	5.32	
														0.0030	5.51	5.38
														0.0029	5.32	
C	2	3	2.78	I	0.0015	2.75		0.0015	2.76		0.0015	2.76		0.0018	3.33	
					0.0014	2.56	2.62	0.0016	2.95	2.82	0.0014	2.57	2.70	0.0018	3.33	3.27
					0.0014	2.56		0.0015	2.76		0.0015	2.76		0.0017	3.14	
		6	5.85	I	0.0031	6.16		0.0029	5.78		0.0028	5.54		0.0030	5.94	
					0.0031	6.16	6.16	0.0029	5.78	5.85	0.0029	5.74	5.61	0.0031	6.14	6.14
					0.0031	6.16		0.0030	5.99		0.0028	5.54		0.0032	6.34	
				II							0.0027	5.34				
											0.0028	5.54	5.47			
											0.0028	5.54				

APPENDIX V (Part 2 Cont.)

RAW ANALYTICAL DATA FOR STANDARD CONCRETE MIXES CONTAINING SLAG AGGREGATE. CEMENT CONTENT UNITS ARE SACKS/CU YD, CALCULATED NEGLECTING Z-CORRECTION DEFINED IN APPENDIX IV

Slag Type	Cement Brand	Nominal Cement Content	Known Cement Content	Operator	One Day Cure Time			Three Day Cure Time			Seven Day Cure Time			Twenty-eight Day Cure Time		
					SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content	SO_3 Content	Cement Content	Average Cement Content
D	1	3	2.88		0.0016	2.52		0.0020	3.13		0.0019	3.01		0.0018	2.81	
				I	0.0017	2.70	2.64	0.0019	2.96	3.07	0.0018	2.83	2.95	0.0018	2.81	2.96
					0.0017	2.70		0.0020	3.13		0.0019	3.01		0.0020	3.15	
											0.0022	3.53				
				II							0.0022	3.53	3.59			
											0.0023	3.71				
		6	6.00		lost during processing			0.0035	6.19		0.0036	6.35		0.0036	6.37	
				I				0.0033	5.81	6.06	0.0037	6.53	6.47	0.0036	6.37	6.37
								0.0035	6.19		0.0037	6.53		0.0036	6.37	
								0.0038	6.75							
				II				0.0039	6.93	6.75						
								0.0037	6.56							
D	2	3	2.84		0.0016	2.92		0.0016	2.91		0.0017	3.09		0.0017	3.09	
				I	0.0015	2.73	2.79	0.0017	3.09	2.97	0.0017	3.09	3.09	0.0016	2.91	3.03
					0.0015	2.73		0.0016	2.91		0.0017	3.09		0.0017	3.09	
		6	5.75		0.0027	5.22		0.0027	5.24		0.0027	5.22		0.0028	5.44	
				I	0.0028	5.41	5.28	0.0026	5.04	5.10	0.0028	5.42	5.29	0.0029	5.63	5.50
					0.0027	5.22		0.0026	5.04		0.0027	5.22		0.0028	5.44	
					0.0027	5.22		0.0027	5.24		0.0027	5.22				
				II	0.0028	5.41	5.28	0.0028	5.44	5.24	0.0027	5.22	5.22			
					0.0027	5.22		0.0026	5.04		0.0027	5.22				
	*	6	6.14		0.0032	6.05		0.0030	5.70		0.0029	5.54		0.0031	5.94	
				I	0.0032	6.05	5.98	0.0031	5.90	5.83	0.0030	5.74	5.61	0.0031	5.94	5.94
					0.0031	5.85		0.0031	5.90		0.0029	5.54		0.0000	0.00	
											0.0029	5.54				
				II							0.0030	5.74	5.67			
											0.0030	5.74				

* Extra mix; not included in data analysis.

APPENDIX VI

ANALYTICAL DATA CONVERTED TO CEMENT CONTENT (sacks/cu yd) FOR STANDARD NATURAL AGGREGATE CONCRETE MIXES

Aggregate	Known Cement Content	Operator	SO_3 Method: 7 Day Cure: SO_3 Fraction	Cement Content	Avg. Cement Content	7-28 Day Cure: SO_3 Fraction	Cement Content	Avg. Cement Content	28 Day Cure: SO_3 Fraction	Cement Content	Avg. Cement Content	Silica Method: 7 Day Cure: SiO_2 Fraction	Cement Content	Avg. Cement Content	7-28 Day Cure: SiO_2 Fraction	Cement Content	Avg. Cement Content	28 Day Cure: SiO_2 Fraction	Cement Content	Avg. Cement Content
Gravel A Series 2	5.98	I	0.0030 0.0029 0.0029	5.42 5.24 5.24	5.30	0.0030 0.0028 0.0029	5.42 5.06 5.24	5.24	0.0029 0.0029 0.0027	5.30 5.30 4.93	5.18	0.0546 0.0535 0.0517	6.97 6.75 6.36	6.69	0.0518 0.0497 0.0500	6.39 5.95 6.01	6.12	0.0598 0.0595 — —	8.15 8.05 — —	8.10
		II																0.0505 0.0514	6.20 6.39	6.30
Gravel B Series 2	5.79	II	0.0029 0.0029 0.0029	5.14 5.14 5.14	5.14	0.0026 0.0026 0.0026	4.61 4.61 4.61	4.61	0.0027 0.0027 0.0027	4.85 4.85 4.85	4.85	0.0377 0.0378 0.0370	5.24 5.26 5.11	5.20	0.0337 0.0351 0.0386	4.47 4.74 5.41	4.84	0.0381 0.0386 0.0344	5.40 5.50 4.67	5.19
Limestone A Series 2	6.01	I	0.0028 0.0029 — — —	5.28 5.47 — —	5.38	0.0031 0.0031 0.0030	5.85 5.85 5.66	5.79	0.0031 0.0031 0.0031	5.82 5.82 5.82	5.82	0.0340 0.0342 0.0328	5.72 5.76 5.48	5.65	0.0359 0.0369 0.0341	6.11 6.31 5.74	6.05	0.0431 0.0443 0.0444	7.52 7.76 7.78	7.69
		II										0.0320 0.0342 0.0324	5.32 5.76 5.40	5.49	0.0323 0.0332 0.0349	5.38 5.56 5.91	5.62	0.0364 0.0360 — — —	6.18 6.10 — —	6.14
Limestone B Series 2	5.86	I	0.0030 0.0031 0.0031	5.35 5.53 5.53	5.47	0.0031 0.0032 0.0032	5.53 5.71 5.71	5.65	0.0029 0.0030	5.14 5.31	5.23	0.0340 0.0336 0.0337	5.25 5.17 5.19	5.20	0.0418 0.0431 0.0426	6.74 6.99 6.89	6.87	0.0330 0.0334 0.0326	5.02 5.09 4.94	5.02
		II										0.0343 0.0361 0.0341	5.30 5.65 5.26	5.40	0.0374 0.0346 0.0392	5.90 5.36 6.24	5.83	0.0341 0.0349 0.0320	5.23 5.38 4.83	5.14
Gravel A Series 3	5.98	I	0.0029 0.0029 0.0028	5.71 5.71 5.51	5.64				0.0033 0.0031 0.0032	6.52 6.12 6.32	6.32									
		II										0.0443 0.0503 0.0471	4.82 6.08 5.41	5.44				0.0478 0.0517 0.0491	5.58 6.39 5.85	5.94
Gravel B Series 3	5.94	I	0.0027 0.0026 0.0026	5.42 5.22 5.22	5.29				0.0028 0.0027 0.0028	5.65 5.45 5.65	5.58									
		II										0.0420 0.0383 0.0409	6.30 5.55 6.08	5.98				0.0392 0.0413 0.0376	5.76 6.19 5.43	5.79

APPENDIX VII
CEMENT CONTENT DATA (sacks/cu yd) FOR STANDARD NATURAL AGGREGATE CONCRETE MIXES[(1)]

Aggregate	Known Cement Content	SO_3 Method				Silica Method			
		7 Day Cure		28 Day Cure		7 Day Cure		28 Day Cure	
		SO_3 Fraction	Cement Content	SO_3 Fraction	Cement Content	SiO_2 Fraction	Cement Content	SiO_2 Fraction	Cement Content
Gravel A Series 3	5.98	0.0029	5.71	0.0033	6.52	0.0443	4.82	0.0478	5.58
		0.0029	5.71	0.0031	6.12	0.0503	6.08	0.0517	6.39
		0.0028	5.51	0.0032	6.32	0.0471	5.41	0.0491	5.85
Gravel B Series 3	5.94	0.0027	5.42	0.0028	5.65	0.0420	6.30	0.0392	5.76
		0.0026	5.22	0.0027	5.45	0.0383	5.55	0.0413	6.19
		0.0026	5.22	0.0028	5.65	0.0409	6.08	0.0376	5.43
Limestone A Series 2	6.01	0.0028	5.28	0.0031	5.82	0.0320	5.32	0.0364	6.18
		0.0029	5.47	0.0031	5.82	0.0342	5.76	0.0360	6.10
		----	----	0.0031	5.82	0.0324	5.40	----	----
Limestone B Series 2	5.86	0.0030	5.35	0.0029	5.14	0.0343	5.30	0.0341	5.23
		0.0031	5.53	0.0030	5.31	0.0361	5.65	0.0349	5.38
		0.0031	5.53	----	----	0.0341	5.26	0.0320	4.83

(1) These data were used for statistical evaluation of the SO_3 and soluble silica methods.